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EXAMPLES AND THEIR SOLUTIONS

PACKINGHOUSE INDUSTRIES
COTTONSEED OIL AND PRODUCTS
MANUFACTURE OF LEATHER
MANUFACTURE OF SOAP

648

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PREFACE

The International Library of Technology is the outgrowth of a large and increasing demand that has arisen for the Reference Libraries of the International Correspondence Schools on the part of those who are not students of the Schools. As the volumes composing this Library are all printed from the same plates used in printing the Reference Libraries above mentioned, a few words are necessary regarding the scope and purpose of the instruction imparted to the students of—and the class of students taught by—these Schools, in order to afford a clear understanding of their salient and unique features.

The only requirement for admission to any of the courses offered by the International Correspondence Schools is that the applicant shall be able to read the English language and to write it sufficiently well to make his written answers to the questions asked him intelligible. Each course is complete in itself, and no textbooks are required other than those prepared by the Schools for the particular course selected. The students themselves are from every class, trade, and profession and from every country; they are, almost without exception, busily engaged in some vocation, and can spare but little time for study, and that usually outside of their regular working hours. The information desired is such as can be immediately applied in practice, so that the student may be enabled to exchange his present vocation for a more congenial one or to rise to a higher level in the one he now pursues. Furthermore, he

wishes to obtain a good working knowledge of the subjects treated in the shortest time and in the most direct manner possible.

In meeting these requirements, we have produced a set of books that in many respects, and particularly in the general plan followed, are absolutely unique. In the majority of subjects treated the knowledge of mathematics required is limited to the simplest principles of arithmetic and mensuration, and in no case is any greater knowledge of mathematics needed than the simplest elementary principles of algebra, geometry, and trigonometry, with a thorough, practical acquaintance with the use of the logarithmic table. To effect this result, derivations of rules and formulas are omitted, but thorough and complete instructions are given regarding how, when, and under what circumstances any particular rule, formula, or process should be applied; and whenever possible one or more examples, such as would be likely to arise in actual practice—together with their solutions—are given to illustrate and explain its application.

In preparing these textbooks, it has been our constant endeavor to view the matter from the student's standpoint, and to try and anticipate everything that would cause him trouble. The utmost pains have been taken to avoid and correct any and all ambiguous expressions—both those due to faulty rhetoric and those due to insufficiency of statement or explanation. As the best way to make a statement, explanation, or description clear is to give a picture or a diagram in connection with it, illustrations have been used almost without limit. The illustrations have in all cases been adapted to the requirements of the text, and projections and sections or outline, partially shaded, or full-shaded perspectives have been used, according to which will best produce the desired results. Half-tones have been used rather sparingly, except in those cases where the general effect is desired rather than the actual details.

It is obvious that books prepared along the lines mentioned must not only be clear and concise beyond anything

heretofore attempted, but they must also possess unequaled value for reference purposes. They not only give the maximum of information in a minimum space, but this information is so ingeniously arranged and correlated, and the indexes are so full and complete, that it can at once be made available to the reader. The numerous examples and explanatory remarks, together with the absence of long demonstrations and abstruse mathematical calculations, are of great assistance in helping one to select the proper formula, method, or process and in teaching him how and when it should be used.

Three of the volumes of this library are devoted to subjects pertaining to Applied Chemistry. The present volume contains treatises on packinghouse industries, manufacture of cottonseed oil and products, manufacture of leather, and manufacture of soap. All these subjects have been treated very fully and every care has been taken to represent the best modern practice. We believe there are no other books that treat on the immense packinghouse industries; that is, on the utilization of those animal products that cannot be used in the form of meats. The cottonseed-oil industry is growing rapidly, and there has been a demand for a treatise that considers the subject thoroughly from a theoretical and practical point of view. The addition of rules and regulations of the different exchanges in regard to cottonseed oil, and the official analytical methods, enhance the value of this treatise. The leather and soap industries are so closely related with the first two subjects that their place in this volume will be fully justified. Both chapters will furnish valuable information to the practical tanner and soapmaker.

The method of numbering the pages, cuts, articles, etc. is such that each subject or part, when the subject is divided into two or more parts, is complete in itself; hence, in order to make the index intelligible, it was necessary to give each subject or part a number. This number is placed at the top of each page, on the headline, opposite the page number; and to distinguish it from the page number it is preceded by the printer's section mark (§).

Consequently, a reference such as § 37, page 26, will be readily found by looking along the inside edges of the headlines until § 37 is found, and then through § 37 until page 26 is found.

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PACKING-HOUSE INDUSTRIES

(PART 1)

INTRODUCTORY

1. Historical.—The packing industry is said to have been carried on in the New England States as early as 1640, but not in the way the modern business is conducted. The oldest record of such an establishment, perpetuated to this day, is claimed by a Philadelphia firm, which started packing in the primitive way of the times in the year 1760 and developed its methods into those of the modern packing house.

The packing industry, with its diversified components, as we now understand it, dates from the year 1818, when an establishment was started in Cincinnati, which city soon became the center of this industry. One reason for this was the surrounding corn-raising area—the natural territory for a steady supply of hogs. As the population and facilities for shipping increased, the corn-belt area moved farther west, followed by the packing houses locating near their supply of raw material. Chicago followed Cincinnati as the center of the packing industry, being situated in the corn-raising area, combined with its natural transportation facilities at the head of Lake Michigan. This city is indisputably and universally acknowledged the center of the meat industries of the world. Thousands of cars of cattle and hogs, both live and dressed, are shipped from there annually in addition to those animals slaughtered for adjacent consumption.

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Within the last few years, large and important interests have developed in the packing industry still further west, notably in St. Louis, Omaha, and Kansas City, for the obvious reason of the close proximity of supplies of cattle and hogs raised in the sparsely settled Western States. Although these cities are large packing centers, Chicago, mainly owing to its prestige and almost unequaled lake and rail shipping facilities, still maintains its supremacy.

The primitive methods of this business have practically been revolutionized. In former times, natural temperatures and ice were the only means available for preserving the manufactured products, in consequence of which the packing was carried on in the winter months only. Today, artificial refrigeration and the power to control temperatures at will has given this industry the advantage of being able to be carried on daily throughout the year. In addition to this, it is now possible to consume the meat of animals slaughtered thousands of miles distant from the great consuming centers, it being transported by scientifically built refrigerator cars, a thing unknown until a few years ago.

In addition to artificial refrigeration, another most potent factor in developing the modern, scientific packing industry is applied chemistry. Were it not for the ingenuity of the chemist in finding a way to utilize the offal of slaughtered animals, the price of meat would necessarily be very high. The chemist has turned into practical channels of income what was in former times not only waste, but a source of expense for its removal.

The object of the primitive packing industry was to produce meat. The object of the present scientific packing industry is not only to produce meat, but also to utilize every available part of the animal, which objective point is attained to the highest degree in the large modern packing house of today.

2. Breadth of the Packing Industry.—This subject is so very broad that today it covers an enormous field. Not only does the modern packing house pack pork and beef similarly to when this industry was started on a large scale,

but it also produces and handles many foods other than those produced from live stock.

The modern packing establishment may be termed, in common language, a *food factory*. Animal and vegetable foods and compounds of the two are produced in many forms; together with these there are also produced other edible compounds, which closely verge on the pharmaceutical field. This is evidenced by the production of beef extract, which may hardly be considered a food, but rather a stimulant, as prepared in the packing house.

A step further than this we have the preparations known as *digestive ferments*, not food, but still alimentary products. Still further bordering on to the pharmaceutical field are the preparations frequently produced (in the largest establishments), such as *inspissated ox gall* and the so-called animal extracts, as *cardine*, *testine*, *cerebrine*, etc.

3. There are so many offshoots in the packing industry today that it is difficult, in fact, to draw a line where this industry ends. So many by-products of the animals slaughtered are now manipulated into finished products by the packers that the whole tendency is to handle in one establishment the entire product and by-product of the slaughtered animals. This is evinced by one of the largest packers converting every by-product of hogs, sheep, cattle, and calves, with the single exception of hides, into manufactured finished products. For example, glue, sizing, hair felt, soap, anhydrous ammonia, pepsin, pancreatin, curled hair, tankage, and the finished, complete fertilizer are now made in one establishment.

The meat-canning industry is closely associated and almost always contiguous to the packing house, both for convenience and profit. This industry is a complete one in itself, although practically inseparable from the packing-house industry proper. The canning industry will therefore be described, as the economical management of the modern packing house depends to a very large extent on this resourceful branch of the business.

The most important feature in the utilization of by-products in the packing house is observed in the fertilizer department, as here all animal products not useful for food or manufacturing purposes are converted into a remunerative article—fertilizer—either finished, i. e., complete fertilizer, or the crude form, known as *tankage*. This will be treated in its proper place.

The establishing of this department may be said to mark the transition of the simple slaughter house of former days to the modern packing house in all its phases. Formerly dressed meat only was produced, and the offal was a source of expense and encumbrance to the packer, who willingly paid for having it taken away. With the knowledge acquired from the utilization of one by-product, the utilization of all followed as a natural consequence.

The founding of the glue department by the packing houses as a necessary adjunct is among the most recent innovations and at the same time one of the most important. This subject will be treated at length under its proper caption, as will also various other closely associated industries.

4. The development of the packing industry that we have today is due in no small measure to the knowledge and research of the packing-house chemist. Not only has the chemist aided this development, but even today is engaged in furthering the utilization of the various waste products. In addition to this, the modern packer depends on chemistry to aid him in conducting the whole industry on an economical and profitable basis.

This is readily apparent when the many operations are described in which chemistry plays such an important part in determining the grade or class and, consequently, the price of the finished product. The chemist to the packing house is daily called on to perform tests and analyses by which each day's output is controlled. For example, the amount of tallow or grease remaining in the cooked offal is such an important feature that a daily report on the percentage left in this material is given to the superintendent.

This one feature alone may be the cause of profit or an immense loss to the establishment, and thus shows the importance of applied chemistry in this industry.

5. Method of Conducting Establishments.—The modern large packing house is conducted on strictly business principles in every department. The different departments are regarded as independent concerns, the raw material received by each department being considered as purchased from an independent source, and the cost price charged up to its account. At the same time, the department furnishing the material is credited with the price charged. The finished product turned out by each separate department when sold has the amount received credited to that department. All supplies, labor, utensils, etc. constitute a direct charge to the department receiving them; and, in addition, to each is apportioned, pro rata, insurance, taxes, cartage, steam, light, office, selling expenses, etc. In this way it is ascertained what each department is contributing to the general profit or loss. Each day a report of its receipts, shipments, labor, etc. is given to the general manager, who, in the summary, is thus enabled at a glance to see the status and condition of the whole establishment. The labor cost is closely scrutinized, and should it be found that this is in excess of the regulation established, an investigation immediately results and an explanation obtained. From years of experience and by frequent tests made in all departments of the establishment, the exact cost of each product and by-product is accurately known and regulated.

Constant investigations, with a view to perfecting methods and curtailing wastes, are being made, and in this way new avenues are found for the still further profitable working and disposition of by-products.

6. In no business is there more attention given to detail, and probably there is no business where the average profit on each individual product is of such slight margin that the slightest inattention to any detail results in such serious losses. There have been periods in the packing industry

when operations have been carried on in almost every department at a positive loss; but as so much machinery and apparatus are required in this industry, the loss incurred from operating at a small loss was far less than would have occurred from idle machinery, rusty tanks, etc. It may be truthfully said that of all departments of this industry there is but one from which a good profit is always expected and most always obtained—namely, the sausage department. This has the advantage of being able to utilize parts from all slaughtered animals, while the other departments manufacturing the goods are restricted to a particular class of material.

The general methods of treating and caring for packing-house products and by-products will be presented as they are daily carried out in the modern packing houses in this country.

VARIOUS ANIMAL PRODUCTS AND THEIR DISPOSITION

BEEF PRODUCTS

7. General Remarks.—The disposition of a bullock in the modern packing house is quite complex. To obtain the best financial results, the operations of caring for the various by-products are carried out in great detail. By reference to the chart, Fig. 1, it may be seen into what products a bullock is ordinarily resolved. It is important for a chemist dealing with animal products to know the source and nature of material with which he has to deal. Further than this, knowing the characteristics of certain products, he can very often, by a superficial examination of material in question, at once name the source of its derivation.

This is especially true of tallows and lard. Certain parts of animal fats have, as is well known, different melting points. For example, the tallow from a bullock's head will have a titer, or hardness, not exceeding 41.5° or 42° C., while

the tallow from the small intestines will have one of about 44° C. When an ordinary tallow is found with a hardness of over 44° C., the origin is at once placed to the fat of the the kidneys.

The resolution of the bullock will be treated first, that of the hog following. While the chart, Fig. 1, shows the detail in a general way, some of the by-products are still further subdivided. The method of handling in the particular packing house determines this. For example, as shown in the chart, the feet, among other things, yield sinews, neatsfoot oil, and raw bone. Taking these products, we may obtain, by further treatment of sinews, the products glue, neatsfoot oil, and tankage. From the neatsfoot oil may be obtained cold-pressed neatsfoot oil and neatsfoot stearin. From the raw bone may be obtained glue, tallow, and bone meal. So while the chart represents approximately the ultimate disposition of the by-products, it must be remembered that further treatment is frequently practiced. Further, not all these many by-products are made in all establishments. As the present conditions in this business, however, demand the utilization in the same plant of everything possible, the treatment of each product will be described.

8. Hide.—This is removed from the freshly slaughtered bullock, and after being trimmed free from adhering flesh and fat, is allowed to cool for at least 24 hours to eliminate the animal heat. If this were not done and the hide salted while more or less warm, decomposition would ensue to some extent, which, while held in check by the salt, would so injure the fiber of the hide that the tanner would be unable to make good leather from it. The hides each have about 25 pounds of salt spread over them and are piled, one on top of the other, to a height of about 3 feet. They are allowed to remain in this condition until cured, which generally requires about 3 weeks. The hides are then termed *green-salted hides* and are sold in this condition. Several grades are selected in the packing house, varying according to weight, brand, size, and general condition.

9. Head.—After removal from the body the head is trimmed free of meat, which is utilized in the making of sausages and for canning. It has the horns sawed off, if there be such on the head. Horns constitute a very profitable by-product of the packing house, as they bring good prices. Inside the horn is a tough, porous, semibony filling termed *horn pith*, which is an excellent material for the making of high-grade edible gelatine. The piths are removed from the horns by means of hot water and dried carefully.

The head is then separated into the skull and the jaw bone. The brain is removed from the skull, if desired for edible purposes, and the latter is then placed in a vat of hot water for cooking. The jaw bones are cracked across, to enable the contained tallow to be boiled out in the vat, in which they are placed with the heads. They are washed, to remove the adhering blood and dirt, and boiled with live steam for about 10 hours. This treatment gives tallow, which is removed from the top of the liquor; and a watery solution of glue, known as *glue liquor*, the treatment of which will be described later.

The bones remaining in the vat are freed from adhering meaty matter, washed, and dried on steam coils. They may be disposed of in this condition or they may be crushed by a bone crusher and made into crushed raw bone, or the latter may be put through a bone mill and ground into raw bone meal. An analysis of this will show, approximately, ammonia, 5 to 5½ per cent. and bone phosphate 55 per cent.

The meaty material from the heads and the small bones left behind, are then put into a rendering tank, to obtain whatever tallow may be left in them. The tallow obtained from heads is frequently called *butter stock*. It has a titer test of about 41.5° C., the free acid, if the heads have been cooked fresh, not exceeding .5 per cent. This tallow is frequently used for the making of oleo oil and for mixing with lard.

10. Food Products.—Although parts other than these are used for food, the liver, heart, tail, and tripe may be

conveniently grouped under this head. The liver taken from the bullock has the gall bag removed and is then washed and sold in the fresh state. Where a surplus of livers are obtained, they are tanked for fertilizer material, making tankage, which will analyze about 15 per cent. ammonia and no bone phosphate. Livers when cooked yield no tallow.

11. Heart.—After removal, the heart is cut lengthwise to a slight extent, to allow the contained blood to escape. It is then washed in cold water and when thoroughly chilled used for various purposes. Among these are for canning, mixed with other meat, for pickled goods, similar to pig's feet, and very largely for the manufacture of beef extract. This process will be described later. Hearts are also sold in the fresh state for food.

12. Tail.—The tail is chilled after removal from the bullock and is sold fresh, or utilized for the making of the canned soups. As the tails contain a large amount of gelatinous matter, they cannot be used for beef extract. With these, as, in fact, with all surplus of packing-house by-products when beyond the proper condition for food, the fertilizer tank is their ultimate disposition.

13. Tripe.—Tripe is made from the first stomach or paunch of the bullock, containing the undigested food. The latter is emptied and is utilized for the making of strawboard. This use at present is somewhat limited and by no means utilizes all this refuse material. The large packing houses pass this, when not desired for strawboard, through large revolving rollers, pressing out the surplus water and juices, obtaining a material carrying about 50 per cent. of moisture. This is then mixed with coal and used as fuel. The fertilizing value of this paunch manure is very slight, as it contains when dried but 2 per cent. of ammonia. Its low gravity and bulkiness make it undesirable for fertilizer mixtures.

The paunch itself is washed in ice-cold water and trimmed free of adhering fat. The latter when rendered makes a hard tallow having a titer of about 44° C. and has always a greenish-yellow color. The free fatty acids in this tallow are about 1 per cent. The material is used either for oleo oil or for mixing with other common tallow. The cleaned paunch is cooked in hot water at a temperature just under boiling for about 2 hours and then scraped free of the outside skin. It is then placed in vinegar and sold as tripe. Tripes when tanked yield $2\frac{1}{2}$ per cent. of tallow, $7\frac{1}{2}$ per cent. of dried tankage, and about 9 per cent. of dry glue, obtained from the glue liquor. The tankage will analyze, on the dry basis, ammonia, 15 per cent.; ash, 7.65 per cent.; and fat, 4.4 per cent. Tripes are seldom used as a source of glue material, owing to their greater trade value in other channels. Experiments have been made to convert these into leather, but without much success.

14. Beef Gall.—This product, known in pharmacy as *fel bovis*, is frequently manufactured into inspissated ox gall. For this purpose, the gall is collected from the gall bladders and evaporated in an ordinary steam-jacketed kettle until a thick skin forms on the top of the thick liquor. The original bulk is reduced about three-fourths, when it is poured into shallow sheet-iron pans about $1\frac{1}{2}$ inches high, 30 inches long, and 20 inches wide. At a temperature of about 212° F., it is evaporated to dryness and kept in the drying room until wanted for grinding. As this material is very hygroscopic, this precaution is very essential.

The grinding, or powdering, is accomplished in a specially constructed mill, grinding out of contact with the atmosphere. Any machine that fills this condition is suitable for this purpose. The hard and brittle dried gall is removed from the pans only when wanted for immediate grinding. When sufficiently fine, the gall is removed from the mill and at once placed in tin cans, which are immediately sealed air-tight by soldering.

The inspissated ox gall is used to a considerable extent for medicinal purposes. The liquid gall has been employed in the manufacture of special soap for washing goods the dyes of which are loosely fixed. The liquid article is used to a limited extent in fixing the dyes and colors of fabrics and carpets.

15. Feet.—These comprise that part of the legs from the knee joints to the hoofs. After removal, they are stripped of the sinew lying along the shin bone. The sinews are, in most packing houses, salted down and held in this condition until sold to the glue manufacturer. They may be cooked by themselves in the packing house, when they are resolved into glue liquor, yielding about 20 per cent. dry glue, 5 per cent. neatsfoot oil, and 3 to 5 per cent. dry tankage (not including the large bones of the feet).

The feet are next passed to the sawyer, who removes the knee joint and the foot proper with the attached hoof, in both cuts exposing the marrow at the ends of the shin bone. The feet are then placed in boiling water for 15 minutes, removed, and the hoofs forced off by a machine, leaving them in condition to be boiled for oil, glue liquor, and bones. The hoofs are then either dried to be sold in this condition or are made into hoof meal, which is described later.

The feet are cooked in open vats with live steam for about 10 hours, or until the bones of the feet fall apart and the oil separates. The glue liquor formed is cared for by the glue department, when in connection with the packing house, or may be concentrated to a jelly and sold in this condition to the glue manufacturer. The bones, washed free of adhering meaty matter, are dried in this condition as raw bone, or may be ground into raw-bone meal, as will be described further on. The ends of the sawed shin bones are boiled with the feet, as they yield the same products.

The tankage or refuse from the boiled feet is generally recooked for several hours, to obtain a further yield of oil. A set of feet (four) give approximately half a pound of dried,

high-grade tankage. The oil that exudes from the feet in boiling is neatsfoot oil, and is collected from the vat, freed roughly from water and scrap by settling, and then placed in an iron tank and heated to 220° F., to drive off all contained moisture. The oil while hot is passed through cotton-flannel bags or, where in large quantities, through a filter press, to remove fine scrap, hair, and other extraneous impurities. The oil when cool is ready for sale purposes, as pure neatsfoot oil, or may be further manipulated, being made into neatsfoot, stearin and winter-pressed neatsfoot oil. The latter process will be described further on.

The sawed shin bones are also boiled in open vats with live steam, to cook them for cutlery bones; from the contained marrow is obtained neatsfoot oil also, which is mixed with that from the knuckle bones of the feet. The refuse from the meaty matter of the bones is sent to the fertilizer department. The bones destined for cutlery purposes are cooked 5 or 6 hours at a temperature not exceeding 210° F., in order to retain as much of the gelatinous matter as possible, thereby keeping the strength of the bone more or less intact. It is important not to overcook these, as by so doing their value is reduced to that of raw bone. In proper condition after cooking and drying, these bones are worth from \$50 to \$80 per ton. The cooked bones, freed from oil and tankage, are dried on racks at a moderate temperature, about 70° F., and are then ready for shipment.

Where these shin bones are not handled in this manner, if not desired for fancy bones, the whole shin and foot, denuded of the hoof, is boiled together for the production of neatsfoot oil and raw bone, the tankage, as usual, being incidental. Raw bones of this description furnish a most excellent material for glue making.

16. Manufactured Goods.—These include material, always, even in small establishments, finished in the packing house. The most important of these articles are sausage casings. They, primarily, are the intestines of the bullock, and are treated most systematically, to obtain as clean

and sterile a product as possible. Casings are divided into three kinds, known as *rounds*, *middles*, and *bungs*. The latter is the large intestine of the bullock connecting the stomach with the smaller intestines, varying in length from 3 to 8 feet, and from 4 to 5 feet when cured (salted). Beef middles, or casings, are the large intestines of the bullock. These vary in length from 20 to 38 feet. This intestine lies between, and is connected with, the bung gut and the round intestines, or guts, as they are termed in the packing house. The round gut is the long intestine of the bullock, and varies in length from 100 to 140 feet.

These intestines lie connected with a mass of fat, the latter being a heavy, apron-like mass called the ruffle. These intestines are subjected to thorough cleaning after separation from the fat, both inside and out, to remove contained manure and adhering slime. The cleaning of intestines for the manufacture of sausage casings depends wholly on removing the impurities from the membrane, and is purely a matter of physical manipulation. The intestines after cleaning either by hand or by a casing-cleaning machine are covered with salt and bundled together for a few days, to become cured or impregnated with salt. They are then shaken free of salt and resalted with very fine salt and packed in barrels and kegs for sale.

17. Weasand.—This is the windpipe or lining of the throat of the bullock and is a tough, translucent membrane. It is surrounded or covered with a layer of dark, soft meat, which is stripped off and utilized for the manufacture of beef extract or for sausage meat. The weasands are then blown full of air and tied tightly. In this condition they are dried for 24 hours at a temperature of 130° F., with a 2-pound weight hung to each one, when they are removed from the drying room, one end cut off, and flattened out. They are then bunched in bundles and sold in this condition. Both wide and narrow weasands are made, the former being blown to their full capacity and the latter but half distended with air.

18. Bladder.—The bladder is freed from urine, washed, and trimmed free of adhering fat, the latter being utilized for tallow. The bladder is then blown to its fullest extent with air and tied round the neck with a string while in this condition. It is dried at the air temperature, the neck cut off, flattened out, and sold in bunches of dozens. Bladders are used extensively for the packing of putty.

19. Fat.—From the bullock three grades of fat are obtained. The best grade comprises the abdominal fat or caul fat, the ruffle fat, and the fat from the top part of the heart. This is all known as No. 1 fat, and is used for the manufacture of the best grades of oleo oil and oleo stearin, the manufacture of which is described later.

The next grade is fat, clean, but in small pieces, and mixed with other material, as meat and bones. This fat is washed in the tank for rendering and makes the ordinary tallow of trade. This tallow may be further separated into tallow oil and tallow stearin, the manufacture of which is also described later.

The lowest grade of fat is that intimately mixed with dark-colored offal or material that, when cooked, imparts an unremovable dark-greenish color to the resultant tallow. This rendered tallow is known as No. 2 tallow, and is always of dark color and strong, fetid odor. The free fatty acids in this material may reach 30 per cent., but usually are in the vicinity of from 12 to 18 per cent. Beef material and offal of no use for anything but fertilizer, together with cooked livers and lungs, furnish this grade of tallow.

20. Horns and Horn Plths.—Among the important by-products of the bullock are the horns. The constantly growing practice of dehorning has caused the supply to diminish from year to year, and good horns are becoming scarcer each year. In spite of this fact, the value of horns has not been increased to any considerable extent, owing to the fact of manufacturers using celluloid and similar compositions as horn substitutes.

21. The horns on the cattle heads are sawed off close to the skull after slaughter. To separate the enclosed pith in the horn, it is only necessary to cook the whole at 160° to 170° F. for thirty minutes. The hot water is then replaced by cold water, to cool the horns and make their handling easier. A heavy stroke of the horn on iron, or vice versa, will cause the pith to fly out. It is very important that horns should not be overcooked, as this will cause them to turn yellow and become brittle, thereby seriously damaging them.

After the pith is separated from the horns, the latter are sorted into various grades—steer, cow, bull, stags, stumps, and useless horns. *Steer horns* are the most valuable, and have a smooth surface on the outside as well as on the inside. Their weight is small considering the size of the horns, varying from 40 to 100 pounds per hundred pieces. They are classed as No. 1 horns. All steer horns averaging below 40 pounds per hundred, and cows, bulls, stags, and stumps are termed by the manufacturers as No. 2 horns. *Stump horns* are those of all grades from which the tip is absent or badly damaged. *Stag horns* are of a rough nature, weighing about 2 pounds each. Useless horns include all misshapen, overgrown, or irregular horns.

Horns will lose in drying 12 per cent. of their original weight. They are stored in a cool place, to prevent their becoming too brittle. Horns are used for the manufacture of imitation tortoise shell, for combs, buttons, and similar articles. The refuse horns, clippings, etc. are made into a fertilizer material called *hoof meal*. This is described further on.

22. **Horn piths**, after being knocked from the horn, are stripped of the skin covering them and dried on the floor at a moderate temperature. The cleaner this product is and the freer from bloody tips, the higher price it will bring. Horn piths are used very largely for the manufacture of edible gelatines and furnish the glue maker with an excellent raw material.

23. Blood.—The blood from the bleeding bullock is conducted to a reservoir, from which it is pumped to the cooking tank or vat in the fertilizer department. The liquid blood from the average beef is 40 pounds. The treatment of this is described later.

24. Fertilizer Materials.—All material from the slaughtered bullock not wanted or not suitable for other purposes may be classed under this head. It includes lungs, pecks (the third stomach of the bullock), spleen, and refuse from the manipulation in the packing house of the other by-products. This material, when rendered, furnishes tannage, tallow, and tank water, the latter being the cooking water heavily impregnated with nitrogenous compounds. Tank water is at the present time made into a fertilizing material by evaporation, known as *concentrated tannage* or in the packing house as *stick*. The treatment of this is described further on.

The above-named products, as before mentioned, may be, and are, subdivided in many cases, but this subject is best treated under the different classes of products, where they are discussed more fully.

HOG PRODUCTS

25. General Remarks.—The dressed carcass of the hog is capable of many dispositions, which depend on many conditions, notably the locality for which it is destined. The various methods of transforming the dressed hog into the very numerous cuts, regulation and otherwise, is so complicated that personal observation and practice is far more valuable than description. The resolution and disposition of the offal is more complex than that of the bullock. The offal from the hog, speaking comparatively, is of more value than that of the bullock, as the finished by-products of the former are much more remunerative to the packer than those from beef.

The chart, Fig. 2, shows, approximately, the resolution of the hog into the most important packing-house products. The same is true here as with the chart of resolution of the bullock; that is, while showing the general result, many of the by-products may be still further divided when deemed profitable. In every establishment, all these by-products are not manufactured, but in the largest packing houses, which are conducted on scientific principles with the aid of chemistry, these, together with many allied products, are manufactured daily in very large quantities. As many packing houses handle hogs only, the utilization of every product possible under the circumstances is a necessary factor in obtaining satisfactory financial results, the latter being the basis on which this, as well as all industries, is conducted. The disposition of the offal in the packing house will be described in detail.

The most important by-product, although not always considered as a by-product, is lard. This subject is so important and the treatment and manufacture so varied that it will be treated under a separate heading.

26. Head.—The head, after removal at the time of slaughter, is thoroughly washed in water, to remove the blood and adhering dirt. Several dispositions are made of this product. In all cases, the tongue is removed and treated by itself. If the heads are intended for sale purposes, they are scraped free of all hair and bristles, washed thoroughly clean in cold water, and chilled for 24 hours, when they are ready for sale. At times, they are split in halves and pickled in strong brine for 2 weeks, when they are packed in barrels for shipment, mostly to tropical countries. When intended for lard purposes, the heads, after being cleaned and washed, are sent to the rendering tank with other suitable material and cooked for lard. Still another use for the heads is the manufacture of headcheese in the sausage department.

Here they are cooked in open vats until the flesh loosens, when they are removed and the bones separated from the

meat. This, together with the skin of the hog or similar glutinous matter for adhesive purposes, is made into a chopped mass with other materials and stuffed into cleaned hog stomachs. This latter use is the most profitable for the packer, as by it he obtains lard from the cooking of the heads, the meat for a profitable use, and the bones for the fertilizer.

27. Blood.—The blood from the slaughtered hog is conducted to a reservoir and treated the same as the blood from cattle, as will be described.

28. Food Products. — From the hog, this broadly includes almost every part of the hog. With the exception of the gall, bones, and bristles, all parts may serve as edible products. Specifically, they will be treated here as the heart, liver, and leaf lard, the latter finding its most important use as neutral lard and as a component of oleomargarine, or butterine.

29. Heart.—The heart finds its most important use in the production of sausages. It is frequently sold attached to the liver and lungs by a short piece of the windpipe, the whole being technically termed the *pluck*. When not desired for these food purposes, it is sent to the tank and rendered for fat, the fleshy residue furnishing a fertilizer material rich in nitrogen.

30. Liver.—The liver of hogs in this country finds restricted use as food, but in many foreign countries this is their only utilization. The fresh livers, preserved in a solution of salt, borax, and boracic acid, are sent abroad from the large packing centers, arriving at their destination many days afterwards in a condition hardly distinguishable from the liver freshly removed from the hog.

The use of hog's liver as a component of piquant table sauces is, although limited, very serviceable. They are also cooked, dried, and then ground into a powder and used as a component of dog biscuit. This outlet is a very important factor at times in disposing of a surplus. When not

desired for other purposes, the livers are cooked under pressure, when they yield only tankage and tank water. The tankage from livers contains about 14 per cent. of ammonia when dried and furnishes a readily and easily available source of nitrogen in fertilizers.

31. Leaf Lard.—Leaf lard when removed from the hog is chilled, as described later, when intended for the making of neutral lard. For sale purposes, it is hung from the middle of the piece, on slanting pegs about 6 inches long, and allowed to chill in this condition. When removed, the skin side exposed, it presents a smooth, cone-like homogeneous lump.

32. Feet.—The feet after removal from the dressed hog, which has been chilled for about 48 hours, are sometimes sold in the fresh state, after being thoroughly cleaned and freed from all hair and the horny toes. The latter are sent to the fertilizer after being cooked under pressure, to loosen the tissue, so they may be easily ground up, furnishing a fertilizing material containing about 19 per cent. of ammonia. This material is generally mixed with the ordinary tankage from hog offal, to raise the grade in ammonia. The small projections on the back part of the feet, called the *haws*, corresponding to the dew claws on cattle, also have their horny covering removed, the material likewise furnishing fertilizer stock. The feet are also cleaned and cooked for 2 hours, after which they are split and placed in packages with vinegar and spices. For this purpose, the feet are first subjected to pickling in a solution of salt and water for 2 or 3 weeks. They are sold as pickled pigs' feet.

When not desired for edible purposes, the feet are cooked under pressure for obtaining lard and tankage. This lard is of a very oily nature, consisting of a large per cent. of olein and very little stearin. It may be mixed in cool weather with ordinary lard, when its oily characteristics are not apparent. Pigs'-foot lard furnishes, upon pressing into oil and stearin, a very large yield of lard oil. It is very frequently utilized for this purpose. The yield of lard from

feet is about 16 per cent. The most profitable use of pigs' feet for commercial products is for glue, which product is largely made in the Chicago packing houses.

33. Hair and Bristles.—The hair and bristles of the hog furnish by-products of extensive utility. The bristles are selected as the slaughtered hog emerges from the scalding tub, from along the ridge of the back and the hams. They are selected as to length and color, the black, brown, and white being kept separate. The hair and bristles are almost always sold by the packer to contracting firms at a certain price per hog, the season of the year, by virtue of the condition of the hair and bristles, largely influencing their value. The contractor always takes care of the product. The hair and bristles are spread in fields to dry, when they are packed with salt and brine in barrels and bags and sent to the brush manufacturers. The hair not suitable for this purpose is sold and made into curled hair. The latter is used by mattress manufacturers, and also for padding purposes in horse collars, cushions, etc.

34. Manufactured Goods.—The same may be said of these products of pork as was said of beef. These include, from the hog, sausage casings and bladders. The bladders are prepared by voiding and soaking in cold water and brine for a day, when they are trimmed of fat and distended with air to their fullest extent, and dried at a temperature of 140° F. for 24 hours. They are then ready for use after expelling the air and folding lengthwise. The rough end, where they are tied to retain the air, is cut off before packing for shipment.

Bladders of hogs find extensive use abroad for packing lard for sale purposes. They are at times colored and used for ornamental carnival purposes. In former years, bladders were largely employed for the packing of snuff by the manufacturers. When parchmented by means of dilute sulphuric acid, they are used for the covering of glass-stoppered jars and bottles.

35. Casings.—Hog casings are classed as *hog casings* and *hog bungs*, the latter being the last 4 feet of the intestines. These, again, are graded according to length and condition. They are thoroughly cleansed inside and out and salted to preserve them, the same way as the beef casings. There is a marked distinction between the texture and appearance of the hog bung and the beef bung. The former is of a close, compact, and solid texture, with a white, opaque appearance. The beef bung is of a fibrous, heavily veined structure and is of a yellowish, transparent or translucent appearance. The hog bung is almost universally employed for liver sausages, while that of the bullock is used for bologna sausages.

Hog casings are made from the small intestines of the hog. The preparation of these differs from the manner of preparing the corresponding beef intestines. They are allowed to ferment for a day in warm brine, to loosen the attached mucous coating. This fermentation has been found by experience to be a necessary procedure for thoroughly and, at the same time, more easily removing the mucous membrane and accompanying slime. Another day's soaking in moderately cold water prepares them for cleaning, which, in small establishments, is done by hand; in large ones, by machines. The treatment of the intestines after cleaning is carried out the same as that of the beef casings, as has been described. This intestine of the hog varies in length from 40 to 75 feet, and, unlike that of the beef, is not fat or embedded in fat, but simply attached to it. The yield of casings per hog is about $\frac{1}{4}$ pound, and the use is for high-grade sausages. The refuse and useless intestines are tanked under pressure for fertilizer material.

36. Stomach.—The stomach of the hog when voided, scraped, and cleaned furnishes an envelope for filling with sausage material, such as headcheese. After cleaning, the stomachs are preserved in a strong solution of salt and water until wanted for use.

The stomachs are also the source of a most important product—pepsin. The preparation of this will be described further on. Most packing houses do not make this finished product, but prepare the stomachs by cleaning thoroughly and shipping to the pepsin manufacturer in a solution of borax and boracic acid. When stomachs are not used for the above purposes, they are cooked under pressure for fertilizer material, furnishing tankage similar to tripe from bullocks.

37. Pancreas.—The pancreas, or sweetbread, furnishes the digestive ferment *pancreatin*. This product is also made only by some of the largest packers. In establishments of moderate size, the pancreas are usually tanked with fertilizer material.

38. Fat.—The fat of the hog suitable for lard material is cooked for the production of lard of various kinds. All fresh and clean fat of the hog is suitable for this purpose. All fatty refuse material not suitable for lard is cooked under pressure, producing grease and fertilizer material. Such material is found in the refuse from cleaning intestines, in catch basins, etc.

39. Fertilizer Material.—The fertilizer material of the hog consists of all material not wanted or suitable for food or other purposes. Products that have become unsalable or have deteriorated are also made into this material and the accompanying grease recovered. During the operation of dressing the slaughtered hog, many parts become so soiled as to be unsuitable for meat or lard, and these, together with all meaty and fatty material, are sent to the fertilizer tank. Clotted blood, the spleen, rejected intestines, etc. also furnish this material.

Hogs condemned by meat inspectors, on account of disease or unsuitable condition at the time of slaughter, are also sent, viscera and all, to the fertilizer tank and rendered into grease and tankage. The water in which all meaty

or nitrogenous animal matter is cooked under pressure is known as *tank water*, with the characteristics previously described.

The products of the hog mentioned are the principal primary ones. As with beef products, these may be still further resolved, as, for example, lard and grease into lard oils and stearins. This subject will be treated later.

CALF PRODUCTS

40. When possible, calf products are disposed of in a manner analogous to that of beef. But the by-products of the calf are very limited, as about two-thirds of the live weight is disposed of as dressed meat. Calves are almost always sold with the skin or hide attached to the carcass.

The **viscera** and internal organs of the calf, together with the legs and head, form the by-products. From the **entrails** and **fat** are produced a small quantity of tallow and tankage. No sausage casings are made from the entrails of calves, as their tender condition renders them entirely unsuitable for handling.

The **feet** yield a small quantity of neatsfoot oil, and when utilized for glue furnish a rich material for gelatine.

The **heads** are sometimes cleaned, scalded, and prepared for food purposes. Where this is not done, they are tanked with the fertilizer material. They also furnish a serviceable glue liquor.

Calf sweetbreads always find a ready sale as a table delicacy at very high prices. The **brains** are frequently removed from the heads and also sold as delicacies. The **blood** and worthless offal are made into the usual fertilizers.

The **heart**, **liver**, and **lungs** attached to a piece of the windpipe, together known as the *pluck*, are always sold in the fresh state for food purposes.

Unborn calves, known technically as *slunks*, are skinned, when the hide is covered with hair. The rest is made into fertilizer.

SHEEP PRODUCTS

41. The offal from sheep is not so diversified as that of the hog or bullock, and is not subdivided to any considerable extent. The **intestines** are all utilized for the manufacture of sausage casings, in the same manner as those of the hog and the bullock.

The **hearts** are used for the making of sausages.

The **livers** find a large sale as "calf" livers. A distinguishing characteristic most frequently present in sheep liver is the occurrence of hard lumps, or cysts, scattered throughout the organ. These are never present in the liver of the calf.

The **lungs** are always tanked for fertilizer.

The **heads** are either tanked under pressure or boiled in open vats for the production of mutton tallow, when in quantity. They are otherwise cooked with the rest of the fertilizer material of the establishment.

The **feet** when cooked by themselves yield a dark-red oil, analogous to No. 1 neatsfoot oil. A test made on sheep feet for oil and tankage gave 4.54 per cent. of oil and 24 per cent. of dry tankage of low grade. Unless a large quantity of material of this nature is always at hand, the feet are cooked with other fertilizer material.

The **paunches**, emptied of manure, are utilized for fertilizer material, as also is all other offal not used for other manufacturing purposes.

The **pelts** of sheep, with the exception of very young lambs, known as *spring lambs*, are removed after slaughter. These are either disposed of fresh or are made into packs with salt interspersed between each skin for the purpose of curing them.

Spring lambs are always sold with the pluck attached to the carcass and with the skin or pelt also left on.

Unborn lambs, if provided with wool, are skinned and the remainder cooked for fertilizer. The skin of unborn lambs furnishes a most excellent material for the manufacture of parchment or vellum.

The **tallow** obtained from sheep product is described further on.

MEAT

42. By reference to the charts, Figs. 1 and 2, it is seen that the broad divisions in the packing industry of both hogs and cattle are dressed meat and offal. As the latter term includes, at the same time, meat as well as other things, it is timely to designate what is meant by the term *meat*. The (German) Imperial Health Office, in a pamphlet issued in the year 1899, covers this definition most comprehensively and also gives therein some very recent analyses of the different kinds of edible meats. The following paragraphs are translated from this document:

43. Definition of Meat.—The word meat, in the broadest sense, means all eatable parts of all kinds of animals. In its proper sense, it refers to muscular meat, and in commercial life, it means muscular meat connected with the corresponding bones, muscular and fat tissues, and glands. As meat is also taken, it includes the tongue, the heart, the lungs, the liver, the milk, the kidneys, the brains, the mamillary glands, the stomach, the guts, the udder, certain parts of the skin, especially from the head and from the legs, and also fat and blood. It further comprises all products from eatable parts of animals (meat and fat products) and preparations, like ham, bacon, canned meat, sausages, gelatine, meat meal, meat extract, salted, corned, smoked, cooked meat (boiled, roasted, steamed, broiled), dried parts of meat, and melted fats, like lard and oleo-margarine.

44. Meat as Food.—Meat is one of the most important foods for man. It contains all nutritious matter necessary for the building up of the body, in useful composition and comparatively easily digestible form. The composition and digestibility, and consequently the food value of the meat, is, however, different with different kinds of animals, and varies with the age and sex and the food used for the animals. Good average percentages from a large number of analyses are given in Table I.

TABLE I

ANALYSES OF VARIOUS MEATS

Meat	Water	Nitrogen	Fat	Ash
Very fat ox meat.....	53.05	16.75	29.28	0.92
Medium fat ox meat..	72.03	20.96	5.41	1.14
Lean ox meat.....	76.37	20.71	1.74	1.18
Fat cow meat.....	70.96	19.86	7.70	1.07
Lean cow meat.....	76.35	20.54	1.78	1.32
Fat veal.....	72.31	18.88	7.41	1.33
Lean veal.....	78.84	19.86	0.82	0.50
Very fat mutton.....	53.31	16.62	28.61	0.93
Medium fat mutton..	75.99	17.11	5.77	1.33
Fat pork.....	47.40	14.54	37.34	0.72
Lean pork	72.57	20.25	6.81	1.10
Horse meat.....	74.27	21.71	2.55	1.01

The quality of the meat is further different in one and the same animal, according to the different parts of the body. The best meat, as a rule, is at the hind quarters; the poorest at the legs. The differences are not alone in the taste and digestibility, but also in its nutritious value, and it is therefore justifiable when, especially in large cities, varied prices are asked and paid for the different parts of meat.

PROCESSES FOR THE MANUFACTURE OF LARD

45. General Remarks.—Lard is the rendered fat of the hog. The several grades of lard produced by the packing house are made by quite different processes, and the care of operating depends on the grade of lard, although careful attention to details is in all of the utmost importance. The cheapest grades are *steam-rendered lard*, that is, the

lard extracted from the stock by the direct contact of steam under pressure. *Kettle-rendered lard* is lard extracted in kettles heated externally, and is the highest grade of household lard. *Neutral lard* is made by a more complex process.

The importance and value of this product is so great, constituting as it does the largest by-product of the hog-killing branch of the business, that the methods of production will be given in detail. Many small points, while seemingly of little moment, are of the utmost importance in the treatment of this material. The production of a high-grade article by properly cooking and handling, in the first place, is not only economical and advantageous, but it also obviates the too frequently applied after treatment necessary to make a marketable lard.

STEAM-RENDERED LARD

46. Rendering Tank. — A modern rendering tank is shown in Fig. 3. The body d , the top d_1 , and the conical bottom d_2 are made of steel or iron, riveted. The head a of the manhole is held in place by the clamp b and nut n . The pipe h is the exhaust to the condenser. The steam

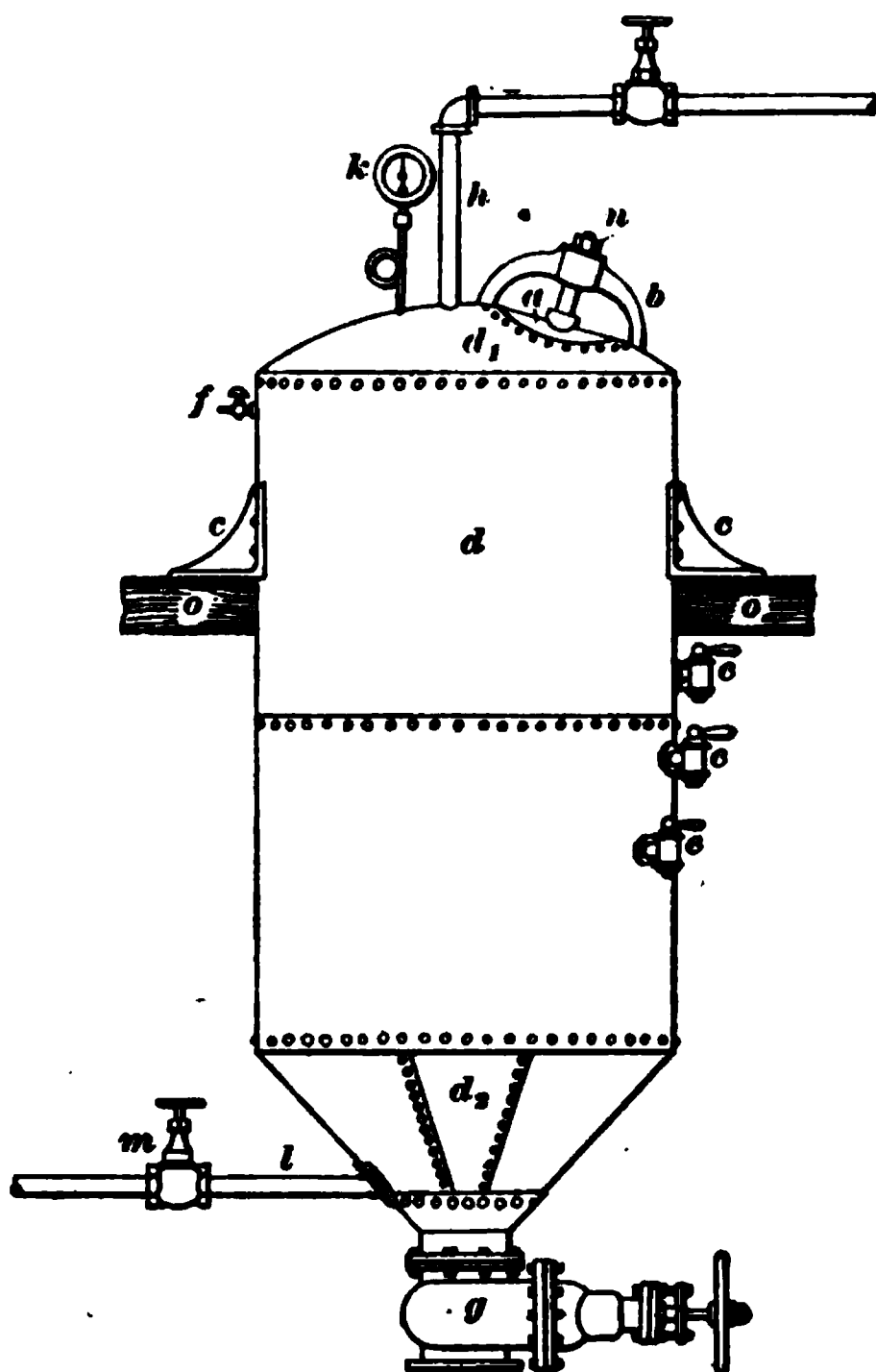


FIG. 3

gauge *k* indicates the pressure in the tank. Steam is admitted to the tank through the pipe *l*, the pressure being controlled by the valve *m*. The valves *c* are for drawing off the lard. The petcock *f* is for the escape of gases during cooking. The gate valve *g* is for the removal of cooked meat, etc.

This is a marked improvement over the old-style tank, which is constructed with cone top and cone bottom. The apex of the bottom cone in such a tank, always below the lowest of any draw-off cock or valve, forms a receptacle for collecting foul drainings from previous renderings and is a most fruitful source of discolored lard or other material, unless great precautions are taken to completely remove this condensed liquid. In the modern construction, this great disadvantage is overcome, as may be readily seen. There are, however, a great many cone-bottomed tanks in use today in packing houses, but they are being replaced constantly by tanks of the newer styles.

Many of the old-style rendering tanks have in them, at the top of the cone or rounded bottom, false bottoms of perforated iron. The object of such a bottom is to keep the raw material from packing below the entry point of the steam, where it would escape cooking to a great extent. Another point with the false bottom is that it allows the water to percolate through it, retaining the solid material on top and level with the door, or *gate*, which, in this style tank is about 15 inches square in the body of the cylindrical part of the tank. Through this gate the solid cooked material is withdrawn by means of long, bent forks. Naturally there is considerable labor attached to this operation, which in the modern tank without false bottom is entirely eliminated. The whole contents of the cooked tank is completely removed in a very few minutes by simply opening the large bottom valve *g*. The saving of this time and labor in the busy seasons in the packing houses is of great importance, as under the old system serious delays were frequent, due to the tanks not being cleansed and made ready for fresh material.

47. The rendering tank is usually 5 to 6 feet in diameter and 14 to 16 feet high. The modern tanks are made of steel instead of iron, and the cylindrical shell is made of two rings. The idea of modern construction is to have as few seams as possible in the whole tank, and to have the laps of the circular rings so that they will point down rather than up, thus preventing the lodgment of corrosive material on the edge of each ring, this material in a short time generating fatty acids that rapidly corrode the seams.

A suitable combination of wood or other material should be on the charging floor surrounding every tank, as by this drippings of water and washings from the floor will be prevented from running down the sides of the tank.

In the old-style tanks, the bottom rested on a timber framework; this proved objectionable from the fact that it prevented access to the bottom plates and at the same time facilitated corrosion by keeping a wet surface constantly in contact with the iron. In modern construction the tank is suspended entirely free from the floors *o*, Fig. 3, except where supported on the lugs *c*. By this method the external surface of the tank is always visible for inspection and kept free from corrosion.

48. Precautions for Safety of Tanks.—Rendering tanks in large cities are subject to the same regulations regarding inspection as steam boilers. Even where municipal inspection is not obligatory, the packers have their tanks periodically tested and inspected. Where the slightest flaw or weakness is detected, the tank is immediately put out of service until made thoroughly safe.

The rendering tank is of such importance in the packing and rendering industries that the greatest care is bestowed on their construction. The life of the ordinary rendering tank is, to a great extent, dependent on the class of material cooked in it and the care it receives in handling. With good usage, the average tank will last about 20 years.

49. Operation of Steam Rendering for the Production of Prime Steam Lard.—The tank, Fig. 3, must be

thoroughly clean on the inside. Where the cone-bottom tank is used great care must be taken to remove all the old tank water or other material from the apex of the cone. The tank is about one-fourth filled with clean water and the material dumped in, care being taken that the fat does not pack where it strikes inside the tank. This is a prolific cause of *cold spots*, and consequently of sour lard. This packing of the fat material as it is dumped in is avoided by distributing it evenly over the interior of the tank by means of a long pole.

The stock in the tank is thus washed while being *loaded*, as the filling is technically termed. To further this washing, a stream of cold water is kept running over the material from a top hose and, at the same time, running out at the bottom at an equal rate, thus maintaining the same level of water on the material. A great deal of blood from the material is in this way eliminated; the more washing lard material receives prior to the cooking, the better will be the quality and color of the lard produced.

When the tank is about three-fourths full of stock ready for cooking, the flow of water is stopped both at the top and bottom and the head put into the tank. This head, similar in shape to a manhole cover, is screwed down tight with a wrench, that should not be longer than 18 inches. When the joint cannot be made tight with that leverage, the head should be removed and refitted with better packing. To force it on with a longer leverage most frequently results in a fracture of the mouthpiece or frame, and when steam is turned on in such a case, the tank is apt to burst and cause loss of life and property.

50. The tank having been properly closed, now has the steam turned on it. This is allowed to enter the tank under full pressure, which is generally from 40 to 50 pounds. The safety valve is now tested to ascertain its free working. This valve should be so regulated that it will blow at the maximum pressure used for cooking the material. The exhaust pipe on the top of the tank is kept wide open until

the pressure gauge shows a pressure of a few pounds, when the exhaust pipe is almost, but not wholly, closed, to allow the steam pressure to rise to the maximum pressure.

The petcock *f* at the top of the tank is always kept open while cooking, to allow the gases generated by the cooking material to escape. These gases are a mixture of hydrocarbons and sulpho-gases, of which hydrogen sulphide forms a large part. These gases, if confined while cooking the lard, would so impregnate it that the lard produced would be of strong and gassy flavor and unsalable as first-class product or *standard* lard in the trade.

When the steam-pressure gauge shows a pressure of 35 pounds, the steam supply is partly shut off, so that the incoming steam is balanced by that escaping from the petcock and the now partly closed exhaust pipe. In this way, the steady pressure desired for cooking is maintained. It is very essential in cooking lard that a steady pressure be held with the steam, as in this way a uniform product can always be obtained.

A tank full of lard material containing from 15,000 to 20,000 pounds should be allowed to cook at 35 pounds pressure for about 11 hours, the time being reckoned from the duration of this pressure. With the above-mentioned quantity of material, the average packing-house tank will be filled three-fourths full of raw material. With the tank only half or quarter full of stock to be cooked, a reduction of time of cooking and also of pressure must be made. With the tank quarter full, a pressure of 25 pounds for 9 hours will be sufficient, while with the tank half full a cooking of 10 hours with 30 pounds pressure will serve the purpose.

51. The object in cooking raw lard material is to break the membranous cells enclosing the fatty material, to allow the oily material to escape from its coverings, and to thoroughly cook the fleshy portions so that what little of this remains in the rendered lard is, in a large measure, prevented from decomposing. Incipient putrefaction of the

raw material is thus prevented, hence the great importance of rendering all material in as fresh a state as possible.

One of the most vital points in making lard is to start with the material in the proper condition, that is, as clean as possible. This applies more forcibly to the lard material from the killing of hogs than to the cuttings from chilled hogs that are made into the customary cuts of hog product for the trade. With stock from freshly slaughtered hogs, it is very important to thoroughly and completely wash it free from blood and the natural filth. If this is not done, the rendered lard will have a dark, brownish color and a disagreeable, strong, pungent odor. Such lard will not pass the usual inspection required for the sale of lard or the requirements of the various boards of trade or chambers of commerce. (See Art. 74.)

52. During the progress of cooking, the operator examines the outside surface of the tank by feeling, to ascertain whether or not the material inside is being equally heated. Should any part of the tank be cold, it shows that the stock is packed solid in that part and the steam is merely cutting a channel for escape through it. This is technically known as a *cold spot*, and, unless remedied, gives rise to sour or putrid lard. The remedy consists in immediately shutting off the live steam and allowing the tank contents to settle for a short time. Then, from the valve in the back part of the tank the water is drawn off until the lard appears. The valve is then closed and the steam again allowed to enter with full force. This procedure breaks up the compactness of the mass and disintegrates the material so that the steam then acts on each piece of raw material. The pressure is then regulated in the manner above described.

It frequently happens that more material is placed in the tank for cooking than should be. In this case, while cooking, the tank becomes *flushed*, that is, the partly rendered lard with the steam and water is blown through both the petcock and the exhaust pipe, causing a serious loss of lard. This is remedied by shutting off the steam and drawing off

from the tank, by means of the back valve, sufficient partly cooked material and water to reduce the level of the contents of the tank below the point where the lard is blown out. The back valve is then closed, the steam again applied, and the cooking proceeded with in the regular way. This is a very common occurrence and is the cause of great losses in this branch of the business, especially in the busy and crowded season, when the tendency is to overload and crowd the tanks, to take care of the large quantity of material.

53. After the cooking is completed, the steam is shut off and the petcock opened wide, to allow the pressure to escape here as much as possible. At the same time the exhaust is closed, as are also the escape valves. The object of this is to prevent the rolling of the contents of the tank, and the consequent forcible projection of the rendered lard through these openings. If there is no sign of rolling, the blow-off and escape valves are opened, a little at a time, observing carefully that the contents do not roll. If the latter occurs, these valves are shut and a short waiting period allowed, until the cooked material has come to rest. After having blown off through the petcock about 30 minutes, the steam is allowed to escape as rapidly as possible, the safety valve being opened to assist in this operation. The object is to reduce the pressure on the tank as rapidly as possible, so that no lard will blow away with the steam.

After the pressure is gone, *and not before*, the head of the tank is loosened and allowed to hang in the manhole. The escape and blow-off, or exhaust, valves are then closed to prevent any induced suction drawing the condensed liquid (made by condensation of the gases) from the exhaust pipe back into the lard. This is a most frequent source of the discoloration of an otherwise perfect lard, and is a point to be carefully guarded against.

54. The rendered lard in the tank is allowed a period of several hours' rest, to enable the fine, cooked fiber to settle with the meaty material in the bottom of the tank. About

10 or 15 pounds of common salt is scattered upon the surface of the lard, the object being to assist, mechanically, the fine scrap to settle. It also serves another purpose: The salt in passing through the lard comes in contact with the water or moisture still saturating it. A union of the water and salt immediately takes place and forms brine, which, being so much heavier than lard, immediately sinks through it, thus eliminating to a large extent the moisture in the lard. The sprinkling of the salt materially shortens the time required for the necessary separation of the rendered lard from the water formed by the steam and the scrap and fine particles of meat scattered through it. The longer the time, within limits, allowed for settling, the better will be this separation and the better will be the quality of the lard when drawn from the tank.

When ready for drawing, or "tapping," the lard from the tank, the water underlying the hot lard is withdrawn through the valve in the back of the tank, near the bottom. This brings the contained lard down to the level of valves *e*, Fig. 3, in the front of the tank. The level of the lard is lowered until, upon withdrawing from the front valves, the lard issues from them clear and free from scrap, when the valve in the back is shut, and the clear lard run from the tank to the usual receptacle. After all the rendered lard has been withdrawn, the lower gate, or drop-bottom, of the tank is opened, and the residue in the tank allowed to run into the vat underneath the tank. This material is then prepared for fertilizer, the treatment of which will be described later. The lard obtained from this process of cooking is *prime steam lard*.

55. Refined Lard.—The lard next in importance to the above in the packing house is refined lard, made from the regular steam lard. Although some years ago the term *refined lard* referred to mixtures of lard with tallow, cotton-seed oil, etc., at the present time in the trade it refers exclusively to the pure and refined steam lard. The process of refining is of a very delicate nature, and outside of the

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FIG. 4

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packing house is unknown in all its details. This process, so important in this industry, will be described in detail. By reference to Fig. 4, the method of operating may be followed. The iron tank *A* is provided with a cone bottom. The contained lard is heated by $\frac{3}{4}$ -inch galvanized iron pipes *c* running around the sides of the tank, the temperature being governed by the steam valve *v*. The blower pipe *x* extends from the blower, or air compressor *Y*, to the bottom of the tank cone, terminating in a circle about 2 feet in diameter. The blower pipe from the top of the tank is 2 inches in diameter. The circle of pipe in the bottom of the tank is perforated with $\frac{1}{4}$ -inch holes on the top, sides, and capped end, for the purpose of distributing the air evenly throughout the lard, thereby agitating it violently when blowing. The cock *m* serves to remove water and settlings from the lard, and is a convenient aid in cleaning the tank.

The siphon *d*, held by the chain *k*, is attached to the feedpipe *e* by a swivel joint inside the tank. The siphon may thus be raised and lowered at will, the lower position representing it when pumping the last portions of material from the bleaching tank.

The pump *F* withdraws the lard through the pipe *e* upon opening the cock *o* and forces it into the filter press *R*, from which the bleached lard emerges and runs through the 2-inch pipe *j* into the agitator *H* for cooling, chilling, and drawing into the requisite packages.

With the above apparatus the operation of bleaching the lard is conducted as follows: The lard is pumped into the tank *A* through the pipe *b* until within about 2 feet of the top. As the lard for this purpose is usually taken directly from the rendering tank, it always contains more or less moisture. The lard is heated to 170° F. and the air blower turned on the lard. This is continued for 15 or 20 minutes, when the moisture will be driven off. If the lard contains much moisture, this will require a longer time. The practical test applied to ascertain the presence or absence of moisture is to fill an ordinary 4-ounce oil-sample bottle with the hot lard and allow it to cool for a few minutes. In the

former case, the lard will become very cloudy and appear thick. When moisture is absent, the clear, hot lard will remain clear for a long period. If the hot lard does not cloud in the bottle within 2 or 3 minutes, no moisture is present.

56. Bleaching With Fullers' Earth.—The bleaching medium is now added to the hot lard. This in packing houses is always fullers' earth. The best temperature for bleaching lard is from 150° to 165° F., depending on the class of material under treatment. The quantity of fullers' earth, or "clay," varies in the same way, from $\frac{1}{2}$ to 3 per cent. being the usual amount necessary to accomplish the bleach. Lard of good grade will require approximately $1\frac{1}{2}$ per cent. to make it snow white.

The necessary percentage of fullers' earth having been added to the agitating lard, the pump is started a few minutes afterwards, the fullers' earth in the interval acting on the lard in absorbing the coloring matter. The lard and clay are pumped into the filter press, the first runnings therefrom being returned to the bleaching tank, as it usually is not quite free from the fine fullers' earth. Until the filter-press cloths become filled and coated with the earth, the lard should be returned to the bleaching tank. The three-way cock *g* attached to the filter-press trough readily allows of this return by running from it through a movable pipe back to the tank.

The thoroughness of the bleach is determined by holding in a water-white glass the bleached lard to the light, when the natural yellowish tinge of lard is apparent. When lard and glass are of the same color, the bleach is perfected, no tinge of color appearing. The lard is then turned through the pipe *j* into the cooler and agitator *H*, where it is stirred and agitated mechanically until cooled to a thick, creamy consistency, so that it is barely able to flow or be drawn off through cocks or faucets.

57. The longer within limits and the more the bleached lard is agitated, the whiter is its appearance in the drawn-off packages. The agitation continues until all the lard

has been withdrawn from the agitator. After the lard has all been pumped from the bleaching tank, the air blower is shut off from the tank and turned on to the filter press to remove the lard held in it between the plates of the press.

The air pressure forces out the lard, which is added to that already in the agitator. To obtain what lard is held by the fullers' earth on the filter-press cloths, the air is shut off from the press and steam forced through the whole press. The lard issuing from the filter press under these conditions must not be allowed to mix with good lard. It is usually sent to the grease tank. The steam is continued on the press until all grease matter has been steamed out. The air now replaces the steam, and the blowing is continued until the cloths and press are dry. After the removal of the fullers' earth from the cloths by scraping or shaking, the press is again ready for filtering.

58. The cloths used for filtering are closely woven strong drilling that is capable of withstanding great pressure. They are usually furnished by the filter-press manufacturers.

The most important points in bleaching are complete absence of all moisture and the proper high temperature—about 165° F. The former condition, however, must prevail, or there cannot be a bleach. When fullers' earth is added to moist lard or oils, it immediately assumes a pasty condition, and when pumped with the lard into the filter press, very shortly clogs the filter cloths with the pasty clay, rendering them absolutely air-tight. When this condition occurs, the whole operation must be started again from the blowing of the material, to eliminate the moisture, but, at best, it is a precarious condition, as too long a contact between the moist clay and lard will impart an earthy taste to the latter, rendering it unsalable.

59. Conditions for Satisfactory Bleaching.—If the temperature of the material is too low, the coloring matter will not be taken from it by the fullers' earth. As a rule, the lower the temperature at which the bleach is carried out, the better will be the resultant product. It is necessary,

however, to have a temperature sufficiently high to cause the absorption of the coloring matter present. So far as chemistry has been able to ascertain, the action of fullers' earth in bleaching lard, oils, etc. is purely physical, no chemical reaction between the two taking place. The physical condition of the earth employed in bleaching greatly influences its efficacy. It has been demonstrated that two clays of the same chemical composition may act radically different in effecting a bleach. One would answer every requirement in this direction, while the other gave no indication of bleaching power, thus demonstrating the fact that the chemical composition plays no part in this process. The fullers' earth purchased in the packing houses is tested in a comparative and practical way, as described further on.

60. Lard Coolers.—Many forms and varieties of coolers or agitators for lard are used. The upright, open tank, in which are revolving arms circulating around stationary arms on a fixed central shaft, is much used. While this will perform the work satisfactorily, its chilling capacity is not sufficient for the largest packing houses with daily outputs of carloads of refined lard. These agitators have a double shell, through which cold water or brine circulates, thereby chilling and agitating the hot lard at the same time.

Another form of a chilling apparatus is a long, semi-cylindrical tank, or box, in which is a revolving, horizontal screw, alternately working in and out the hot lard, exposing it to the air. This cooler sets in continuously circulating ice-cold water. It is adapted only for small manufacturers.

61. Lard Roll.—The most modern, and perhaps the most convenient and economical, method of cooling bleached lard is by means of the lard roll, or cooling cylinder, shown in Fig. 5. These cylinders *a* are made in all sizes, and are well adapted for quick chilling. They are cast in one piece of cast iron and in the refinery are connected with a circulatory system of brine or ice water, thus keeping the surface of the roll constantly cooled. The lard flows on the top of the cylinder as it revolves, and by the time the lard

reaches the attached scraper *c* it is chilled hard. The cooled lard falls into the trough *b* underneath, and is removed therefrom by a pump or spiral conveyer *d* adjacent to an agitator, where the lumps are broken up. The lard is then filled into the various packages from this agitator.

FIG. 5

In some establishments, two rolls are used; one, chilled by ice water, receiving the hot lard, the other, chilled by refrigerated brine, finishing the chilling. Where large quantities of refined lard are made daily, the use of the lard roll is a necessary requisite for rapid working. They revolve at a speed of about ten revolutions per minute and occupy a floor space of about 12 ft. \times 6 ft. Their cost ranges from \$500 each, upwards.

KETTLE-RENDERED LARD

62. Leaf Lard.—This lard is an important product of the packing house, as it is the best grade made for household trade. While this lard is popularly supposed to be made from leaf lard only, it never is made from that alone. The usual proportions of fat from which kettle-rendered lard is made are, approximately, one-third back fat and two-thirds leaf lard, though these proportions are varied at will.

The lard material is filled into a steam-jacketed kettle, which is made to withstand pressure. The back fat has the rind, or skin, removed, and is put in and mixed with the leaf lard. A small amount of fluid rendered lard is put into the kettle before turning on the steam, to assist the material in rendering. The material is cooked until the natural moisture of the fats has been eliminated, which requires about 3 hours for a 3,000-pound batch. The hot lard, when freed from moisture, is quiet on the surface and free from rising steam bubbles. The steam pressure should not exceed 10 pounds per square inch, as shown by the pressure gauge, which gives sufficient heat to cook the lard fully in this time.

The cooked lard is allowed to remain in this kettle until all the fine scrap has settled, when the lard is either drawn directly off through strainers and muslin into packages or run to another settling tank until wanted for filling into packages. The scrap remaining behind in the kettle is sent to the steam-lard tanks for further rendering for any lard remaining in it.

Kettle-rendered lard that has been thoroughly cooked, if free from moisture and scrap, will keep for a long period, even under unfavorable circumstances. This lard, if properly made, does not require bleaching, but, if desired, may be bleached in the usual manner with a small amount of fullers' earth. If the lard becomes burned, or dark-colored material is used, this course is necessary to bring it to the required snowy whiteness.

63. Where large quantities of kettle-rendered lard are made, recourse is had to apparatus similar to that shown in Fig. 6. It is the same in every way, as is the case when making neutral lard, except that the kettle *D* is steam-jacketed instead of being water-jacketed. The lard material is hashed into the kettle and cooked the required time.

Kettle-rendered lard has a characteristic, distinctive, agreeable smell imparted to it by the method of cooking.

Instead of rerendering the cooked scrap, it is often the practice to press this material in a lard press, to recover as much lard as possible from it. The residue remaining is known as *cracklings*, from which the fertilizer material azotine is made. Cracklings are also used for making poultry food by mixing with ground bone.

NEUTRAL LARD

64. General Remarks.—This lard must be what it is named—neutral. It differs radically from other lards, both in its nature and manufacture, and requires apparatus entirely different from that necessary for the production of the other kinds. Neutral lard is made from leaf lard principally, but at times it is profitable to make it from material other than this, which will be described later.

65. Process of Manufacturing Neutral Lard.—The method of producing neutral lard is carried out as follows: The hot leaf lard from the hog is hung, each piece separately, upon flat sticks, fitted into sections in the chill room until the leaf has become thoroughly chilled, which requires at least from 24 to 48 hours. The best temperature for this is just above the freezing point, about 33° F. The leaf lard is then removed to the place of manufacture, which is situated in a part of the establishment remote from odors that might easily impart a taint to this susceptible material and thus render it useless as neutral lard. The apparatus and its arrangement is shown in Fig. 6.

The lard is introduced into the hasher *B*, which is driven by pulley *a* and revolves at a speed of about 600 revolutions a minute. The hasher is provided with a steam jacket through which live steam is constantly circulating during the period of hashing or disintegration. The leaf lard is disintegrated into a plastic mass by the action of the revolving knives attached to the shaft of the pulley *a*, and falls through the spout *s* into the melting kettle *D*. This kettle

is water-jacketed; that is, the inside steel kettle is surrounded by another shell, between which is water, the temperature of the latter being regulated by live steam. The two kettles are not closed at the top, so no pressure or temperature above that of boiling water is possible. The plastic



FIG. 6

lard as it drops into the kettle is constantly stirred by means of the revolving arms fixed to the shaft *c*, which move at the rate of 35 revolutions a minute. The object of this agitation is to prevent the lard from becoming overheated

in any one part by lying against the shell of the kettle. The temperature is gradually raised from the starting point to about 120° F., to overcome the chilling of the melting lard, caused by the cold lard constantly entering the melting kettle. Only sufficient heat is given the water to keep the temperature rising about 1 degree in 5 minutes.

66. It will require about 40 minutes to fill the average kettle, which holds about 3,000 pounds of material. The kettle, filled to within about 6 inches of the top, will be at a temperature of about 126° F. The stirring is continued, keeping the temperature steady at this point for about 15 minutes, when all the lard will be melted. The latter operation is materially assisted by breaking any unmelted lumps with the hands. In about 5 minutes more the fine melted scrap will be seen to collect rapidly at the top, when the operation is completed. The steam is tightly shut off, the paddles, or agitator, raised free from the surface of the lard by means of the chains *m*, and the melted mixture allowed to remain at rest for 15 minutes. The temperature by this time will have risen to about 130° or 135° F., which temperature must in no case be exceeded.

The floating fine scrap from the leaf is then removed from the top, the last particles by a gauze skimmer. The removal of scrap is assisted by scattering about 6 or 7 pounds of fine salt over the surface of the lard. This also facilitates the removal of moisture (see Art. 54). The melted lard is now siphoned off by means of the pipe, with a swivel joint, attached to the side of the kettle; the outside end of this pipe is shown at *e*. The lard as it issues from *e* flows through a fine brass gauze sieve placed over the clarifying kettle *F*, to catch any floating scrap. This kettle is also water-jacketed. The neutral lard is allowed to remain in the clarifying kettle for 2 hours, when it is run through the siphon *g* into the settling kettle *H*, from which, after remaining at least 4 hours, it is drawn into the tierces.

The course of the lard will be followed to the condition required.

67. Packing and Graining.—The lard must be held in the jacketed kettles at a temperature of about, but not more than, 130° F. When drawn into the tierces, it is strained through cheesecloth to catch any possible fine, floating scrap. It should be filled in the tierces at this temperature and at once removed to the graining room, which is kept at as nearly uniform temperature as possible (between 55° and 60° F.). The 3-inch bungs in the sides of the tierces are removed when the tierces are placed on their sides, in order to allow as much lard odor as possible to escape.

After remaining undisturbed for 3 days, the lard will be found to have become of a *grainy* consistency. In other words, the stearin and olein of the lard have separated, and the larger the grains of stearin, the choicer is the lard. After this condition occurs, the tierces may be moved at will; but if disturbed before this separation occurs, the lard will assume the uniform, smooth consistency of ordinary lard and be unsalable. The neutral lard, in the required grainy condition, is then finished and ready for shipment.

68. To return to the melting kettle: After all the clear lard has been removed, the fibrous, brownish residue is let out through the pipe and valve *k* into a receptacle below. This residuum, consisting of water, scrap, and more or less lard, is sent to the rendering tank, in order to obtain, as prime steam lard, whatever has failed to be drawn into neutral lard. An analysis of the scrap removed from the top of the kettle was as follows:

Moisture.....	3.70%
Fiber.....	28.91%
Lard.....	67.39%
Total.....	100.00%

69. Grades and Properties of Neutral Lard.—There are no chemical requirements for neutral lard. The only trade requirements are purely physical, consisting of condition, taste, and complete absence of any odor whatever. The taste must be bland and more or less milk-like. The

condition, as before mentioned, must be a sharp and decided separation of the stearin from the olein. The color of neutral lard is always snow white. The lard made from the leaf is known as *choice, No. 1, or extra neutral*.

70. When the price is suitable and favorable conditions prevail, a neutral lard, known as *No. 2 neutral*, may be made from back fat and fresh ham fat. This material is freed from the accompanying rind, or skin, and treated in the same manner as the leaf lard, with the exception of temperature. This material, containing a much larger proportion of stearin, must be melted at a higher temperature, to obtain a fair yield of neutral lard. The melting of this class of material takes place at a temperature of 136° F., the temperature rising to 142° F., the limit to which it should be carried. The further treatment of settling, etc. is the same as that described for regular neutral lard.

71. Uses of Neutral Lards.—The principal use of neutral lard is for the making of oleomargarine, or butterine. As this lard is not fully cooked, but melted, its keeping qualities are very limited and, hence, no attempts are made to cater to household trade. Neutral lard must always be kept in cold storage, or it will quickly acquire rancidity. The yield that may be obtained from average leaf lard is about 92 per cent. of neutral lard; from the residuum cooked under pressure, a further percentage of steam lard is obtained. The approximate cost for an apparatus to make neutral lard is about \$1,200, and, together with that shown in Fig. 6, consists of a few minor utensils, such as trucks, strainers, etc. Neutral lard always brings a higher price than any other; the greater part of the product is exported, especially to Rotterdam, Holland.

STIFFENED LARDS

72. General Remarks.—In making lard for summer sale or for warm climates, it is of prime importance to make it of such consistency that it will remain in a more or less solid condition. This does not in any way apply to prime

steam or neutral lards, but only to kettle-rendered lard, refined and mixed lard, and, naturally, to lard compound, the latter being made with that object in view, the formula varying with the season of the year. There are several ways of accomplishing the stiffening of lard.

73. Methods of Stiffening Lards.—One method consists in selecting the stock before rendering, by using for steam lard or other purposes the softer parts of the fats, such as feet, and using only those parts that are naturally firm, from the greater amount of contained stearin, for the refined or kettle-rendered lard. While this method is largely practiced in establishments where such selection may easily be made, smaller packing houses are obliged to have recourse to other methods.

A method that is applicable to both large and small concerns and prevails most largely is the addition to the lard of either or both lard stearin or oleo stearin. For producing the desired stiffness, oleo stearin in the proportion of 5 or 6 per cent. is incorporated into the lard while both are in a warm, melted condition. The mixture is kept agitated by mechanical means or by hand until it has reached a temperature of about 110° F., when it will appear cloudy and milky. The lard is kept constantly in a state of agitation, to prevent separation and also to enable it to be withdrawn more freely while thick. The mixture is drawn into the packages as cold and stiff as it can be made to flow from the kettle or agitator.

The filled packages are placed in a cool room, from 38° to 40° F., for 2 days or longer, where there is no possibility of the lard becoming soft. By this time the lard will have acquired a compact condition not easily affected by summer heat. Although it may soften somewhat when subjected to moderately high temperatures (95° to 100° F.), it will never resolve itself into oil and stearin, as will lard that has not been subjected to this treatment. When lard stearin is used for stiffening, the proportion must be increased to 10 or 12 per cent., as the titer of this stearin is seldom over 44°.

Lard stearin added to lard does not come under the head of an adulteration, as it is a natural constituent of the lard itself. Oleo stearin, on the contrary, being a product of beef, is an adulteration. Its use for the purpose of stiffening is quite extensive, the small percentage used rendering its detection more or less difficult.

RULES REGULATING TRANSACTIONS IN LARD

74. The following are excerpts from rules regulating transactions in lard among the members of the New York Produce Exchange, adopted at a meeting of the Lard Trade, held March 27, 1890, and amended March 18 and June 4, 1891, and July 6, 1897.

PRIME LARD STANDARD

RULE 2.—Prime lard shall be equal in quality to lard made from hog round, say head, gut, leaf, and trimmings, in the proportion in which the same come from the hog, and shall be properly rendered as to color, flavor, and soundness for keeping. The renderer's name shall be distinctly marked on each tierce at the time of packing with metallic brand, marking iron, or stencil.

REGULAR TRADES

RULE 3. *Sec. B.*—In the absence of any special agreement, all lard sold on the spot or to arrive shall be understood to be the standard quality of "prime lard," and which is generally termed in option trades, contract lard.

PACKING AND COOPERAGE

RULE 4.—Prime lard made between October 1 of any year and December 31 of the year following only shall be considered "standard," and a good delivery on contracts maturing during that time.

All lard to be classed as "standard" shall be packed in new cooperage made of well-seasoned white or burr oak, free from objectionable sap.

The dimensions of tierces shall be about as follows: 32 inches long with 21-inch head, or 33 inches long, with 20½-inch head; staves to be chamfered at the head; staves ¾ inch thick; head 1 inch thick in center and ¾ inch at bevel; hoops, hickory or white oak, or other good wood, to be hooped not less than 11-16.

STANDARD WEIGHT OF TIERCES AND TANKS

RULE 5.—Tierces shall contain not less than 310 pounds lard nor more than 370 pounds. The "standard" net weight of tierces of lard shall be 340 pounds, and any variation therefrom, when delivered on option contracts, shall be settled for at the settling price of the 11-o'clock call on the day of delivery. The number of packages contracted for must be delivered, and all tierces must have weights and tares marked thereon. Tanks, in the absence of any special agreement, shall be understood to contain 42,500 pounds net, or equal to 125 tierces of 340 pounds each. Any variation therefrom exceeding five per cent. (5%), either buyer or seller may have the right of settling at the market price on date of delivery.

INSPECTORS AND WEAHERS

RULE 6.—All inspectors and weighers of lard for delivery on sale or contract under the rules of the Exchange must be members thereof, and licensed by the Board of Managers, and must obligate themselves not to buy or sell on their own account any article they are licensed to inspect or weigh.
* * * Fees of inspectors and weighers must be paid by the party employing them. * * *

WEIGHT, INSPECTION, AND TARES

RULE 7. *Sec. A.*—The seller shall have the right to designate the weigher, but buyers shall have the right to designate an inspector; either shall have the right to appeal to the committee, as the case may be, whose decision shall be final and binding.

Sec. B.—To determine the tare on lard, four per cent. (4%) of the number of packages shall be tested at the expense of the seller. The tare shall be ascertained by scraping the lard from the packages, and not by removal by dry heat or steam. The empty packages shall then be weighed and the lard replaced, and the weight of the refilled package shall be the gross weight.

Sec. C.—In testing for weight and tares, packages that are evidently mismarked shall be excluded from the average.

Sec. D.—All appeals from weight, inspection, and tares must be settled at the place of delivery unless otherwise agreed upon.

Sec. E.—Seller must give buyer timely notice to attend to inspection, weight, and tares. If buyer fails to attend to the same within a reasonable time, it shall be the duty of any two members of the committee on lard, upon such notice and failure, without fees, to appoint an inspector to sample the lard for delivery on that notice, and his inspection shall be final on that delivery.

LARD CALLS

RULE 9. *Sec. B.*—Unless otherwise specified, all offers to buy or sell shall be understood to be in lots of 250 tierces. Offers to buy or sell in larger quantities than above specified shall be in multiples thereof. * * *

RULE 10.—Either party to a contract, prior to or upon signing the same, shall have the right to call an original margin of two dollars (\$2) per tierce on lard, and a further margin may be called from time to time to the extent of any variation in the market value from the contract price.

Where no original margin has been deposited, calls may be made from time to time to the extent of fifteen (15) cents per one hundred pounds (100 lb.) above or below the market price of lard. * * *

METHOD AND FORM OF CONTRACTS

RULE 11.—The seller shall in every case make out the contracts, and after signing his side shall send them to the buyer not later than the day following the day of the sale. The buyer on receiving the contracts shall sign his side and return it to the seller not later than the second day after the sale.

Verbal contracts, when satisfactorily proven, shall have the same standing as written contracts; but the claim under such contracts must be made on the day of the alleged transaction, or on the next business day thereafter.

The following shall be the form of contract for lard sold for future delivery:

LARD CONTRACT

New York, 18...

In consideration of one dollar in hand paid, the receipt of which is hereby acknowledged, we have this day sold to (or bought from)two hundred and fifty tierces Prime Lard, at.....cents per pound, deliverable at seller's (or buyer's) option.

.....

This contract is made in view of, and in all respects subject to, the By-Laws and Rules established by the New York Produce Exchange in force at this date.

TRANSFERABLE ORDER AND DELIVERY ON CONTRACT

RULE 14.— * * * The lard shall be delivered in lots of two hundred and fifty (250) tierces at one time and one place. There shall not be more than one lot of less than fifty (50) tierces of one brand. Every order must have the weigher's name and place of business indorsed thereon.

* * * * *

APPEAL ON CONSTRUCTION OF RULES

RULE 21. *Sec. A.*—Any party feeling himself aggrieved by the decision of the committee on lard, in the interpretation of these rules, shall have the right to appeal to the board of managers of the Produce Exchange.

Sec. B.—All rules as to lard trades must be justly and liberally construed, and no property shall be rejected or condemned for merely technical reasons, but this shall not be regarded as giving license to departure from their general spirit and intent.

Sec. C.—All former rules of the lard trade, and the general rules of the lard and provision trades, prior to this date, so far as they apply to the lard trade, are hereby repealed.

No change shall be made in the rules by the committee on lard before submitting the same to a meeting of the lard trade, at which ten shall form a quorum.

INSPECTION RATES

At a meeting of the Lard Trade, held May 16, 1892, the following were fixed as the minimum charges on lard handled in New York after this date:

Weighing.....	four cents per tierce.
Inspection and marking.....	four cents per tierce.
Stripping.....	fifty cents per tierce.
Nailing.....	four cents per tierce.

NOTE.—The following addition was made to the inspection rates at a meeting of the lard trade held July 6, 1897:

Inspection and weighing \$5 per tank of 42,500 pounds.

LARD COMPOUNDS

75. General Remarks.—Lard compound and compound lard are trade names by which a material, consisting of no lard whatever, is universally known. Various ingredients are used for making this, the main constituent being cottonseed oil. To this are added, in varying proportions, tallow and oleo stearin; either one may be used or both, in the same compound.

76. Ingredients Used in the Manufacture of Lard Compounds.—The proportion of the different ingredients in

lard compound varies according to the season of the year, the locality to which it is destined, and the market prices of the different components. The formulas most largely used in the packing houses are as follows: For winter compound, cottonseed oil, 80 per cent. and oleo stearin 20 per cent. ; for summer compound, cottonseed oil, 75 per cent. and oleo stearin, 25 per cent. These proportions may be varied at will or according to circumstances. For example, when oleo stearin is high in price, it is replaced wholly or in part by tallow. In the summer formula above, the proportions used could be, with equal results as to flavor, color, and general excellence, cottonseed oil, 75 per cent., oleo stearin, 15 per cent., and tallow, 10 per cent. The same is true of the winter formula.

For warm climates, a summer formula must be used for making the lard compound, while for cold climates the winter formula may be used at all times. The whole object in the manufacture of this product is to make it as nearly as possible like lard in color, texture, and appearance. Naturally, it is not expected to have the taste or flavor of lard and, from the packing houses, it is always sold under its true name, a circumstance, however, that does not always prevail among retailers.

The *cottonseed oil* selected for lard compound should have as good a body as possible; that is, it should contain a notable amount of cottonseed stearin. Cottonseed oils that, on standing in a moderately warm place, deposit a white sediment (cottonseed stearin) should be given the preference over those that, under the same conditions, remain clear. When heavy-bodied oil is used, it requires less tallow or stearin to produce the required consistency, and as the latter products are the most expensive, it is a matter of economy.

The *oleo stearin* employed in lard compound should be selected with as high a titer as possible, for the same reasons as above. The harder the oleo stearin, the more of the cheaper product—cottonseed oil—can be used in the compound. One of the salient features in making this product is to employ as large an amount of cottonseed oil as possible and yet have the resultant product of the required stiffness or consistency.

The *tallow* used for lard compound is ordinarily common, or packer's, tallow. The requirements in regard to hardness are the same for this as for oleo stearin.

While it has been stated that lard is not used in making lard compound, there are no reasons, except that of cost, why it cannot be used as a component. For special trade (export), lard may be used, but the sale price is correspondingly increased. When lard is used for this purpose, the ordinary steam lard will answer all requirements.

Before compounding the different ingredients, they are bleached separately in the usual manner and afterwards mixed in the required proportions.

In bleaching the cottonseed oil, the first step is to ascertain the approximate percentage of clay required to remove the natural yellow color. Some yellow oils are not susceptible of bleach and others require such a large amount of clay that it is both dangerous as well as unprofitable to use them. The danger lies in imparting to the oil the undesirable earthy taste that, once acquired, cannot be eradicated. This test is made in a bottle, as described later.

77. In bleaching cottonseed oil, too high a temperature must be carefully guarded against, as well as the use of more clay than is necessary to accomplish the bleaching. A good temperature for bleaching is about 140° F., although this will vary one way or the other with the particular oil employed. For lard compound, it is not necessary to bleach cottonseed oil water-white, although if possible, and without too much time and labor expended in bleaching, a compound of snowy whiteness is very desirable. The agitation, or sudden cooling, the finished lard compound receives previous to drawing into the packages materially aids in imparting to it the desired white appearance. There being no recognized standard, its usual creamy-white color is acceptable. In bleaching the tallow for use in lard compound, a temperature not over 120° to 125° F. should be the maximum. Good tallows bleach readily at this temperature. It is undesirable, as well as unprofitable, to employ poor

tallow in making compound, as it is very likely to become very strong in taste and smell from the bleaching process, especially where a high temperature has been necessary to effectually bleach it. A small amount of mutton tallow may be used in lard compound, without bleaching, but it is undesirable to use this material, as the strong mutton odor will make itself evident when the compound is subjected to a high heat, as it is in household use.

78. Mixing the Compound.—When all the materials have been bleached separately, they are stored in different tanks, or coolers, until called into use. The proportions of the different ingredients are weighed and united in the mixing tank, from which they are pumped through the filter press and chilled in the usual way, most always, however, over the lard roll. The usual bleaching tank *A*, Fig. 4, conveniently serves the purpose of the mixing tank.

Lard compound, when being drawn into packages, is of such a consistency that a moment after the can is filled it may be inverted and the lard compound will remain in the can unaffected—neither flowing nor dropping out. Of late years, the use of cooking compounds, known under various trade names, as cottolene, cottosuet, etc., have come into existence. These were designed to overcome the prejudice, largely existing, against the name *compound lard*. They are composed quite uniformly of cottonseed oil and beef suet in varying proportions. Many are made in the approximate proportions of 33 per cent. beef suet and 67 per cent. cottonseed oil. While these proportions vary somewhat, they may serve to produce compounds similar in all respects but the registered trade name.

Cottolene is the product of one concern in this country, and its principal distinction from lard compound lies in the fact of its being manufactured from yellow, unbleached cottonseed oil. Recently, this same company has placed a white cottolene on the market. The distinction of this product from the usual lard compound is in its name only.

PACKING-HOUSE INDUSTRIES

(PART 2)

VARIOUS ANIMAL PRODUCTS AND THEIR DISPOSITION—(Continued)

THE FILTER PRESS IN THE PACKING HOUSE

1. General Remarks.—The use of the *filter press* in the packing house is, comparatively speaking, of recent date. It is used for many operations and the preparation of many packing-house products, among them being lard, oils, tallow, glues, and, at times, beef extract. The principal advantages of a filter press are: (*a*) The largest possible filtering surface in the smallest possible space; (*b*) the facility for forcing the material through the filtering medium (cloth) by the most suitable pressure, which varies from a slight pressure to 1,000 pounds working pressure to the square inch; (*c*) the ease with which the filter press may be handled; and, finally, (*d*) the rapidity of filtering large quantities of material at a very nominal cost.

2. Construction of Filter Press.—Filter presses are made with both square and round plates, and of all sizes and capacities. The square press, while not so convenient to handle as the round one, will hold more stock, and is more desirable on this account for packing-house use. The series of round or square plates of cast iron, or other suitable material, are hung upon the press rods. The plates have concave faces on each side, the rim, or outer edge, being

finished smooth and sufficiently wide to avoid unnecessary wear on the filter cloths, and forming tight joints. The concave surfaces of the plates are provided with grooves by means of which the filtered material, after passing through the cloths, passes off. A hole in the center of each plate affords a channel through which the material to be filtered is forced when the press is charged.

Fig. 1 represents a press of 24-inch square plates, which is capable of giving an inch cake (between the plates), and has a working pressure of 150 pounds per square inch. One chamber of this press will hold 484 cubic inches of material to be filtered. A press like this will filter from 7,000 to 8,000 pounds of lard per hour.

Fig. 2 shows a single plate of a filter press, a view of the perforated metal front and also the center clamp being shown. This plate has an outlet cock attached for the filtered material. The plates are covered with the filter cloths, which are placed on each side, are fastened at the center by adjustable screw nuts, and are held in place by

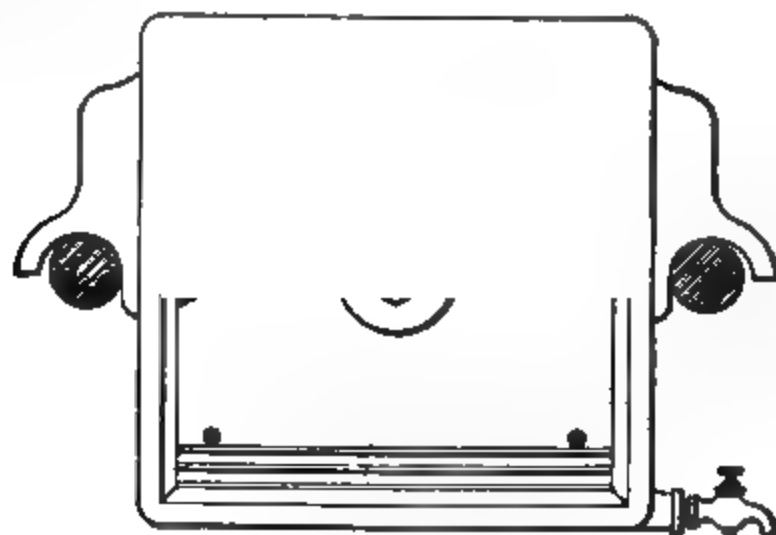


FIG 2

adjustable fastenings, as shown in Fig. 2. When all the plates are clothed, they are forced together by a follower that is actuated by a screw and are tightened by a long lever; or, in some cases, depending on the form of press, by a lever wheel, or by a hand-wheel ratchet lever. Different presses are provided with different means of closing.

3. Filter-Press Cloth.—An important condition in the attainment of satisfactory results consists in the filter-press cloths being of good material. The fabric must be pliable, yet closely woven, so that while giving a clear filtrate, it must be sufficiently strong to withstand the heavy pressure exerted by the pump when forcing the oil or lard through the press. Heavy drilling or cotton duck is suitable for packing-house use.

4. Operation of the Filter Press.—As the pump is applied, the material to be filtered is forced through the center channel, filling all the chambers in the plates. The

pressure forces the liquid through the cloths to the surface of the plates and passes down the grooves, or channels, on the face, through the outlets *a*, Fig. 1, in the plates, into the receptacle, or trough *b*, beneath. The impurities and bleaching material, such as fullers' earth, is retained on the cloths.

The filter press is provided with a safety valve, or outlet *c*, through which, when undue pressure is exerted on the press by the pump, the material may find an outlet instead of bursting either the press or the cloths, as is liable to occur when wet or moist clay is mixed with lard, tallow, or similar material, and efforts are made to force it through the filter press. The valve may be set to operate at any desired pressure; but for packing-house work, 150 pounds per square inch is sufficient. Some forms of filter presses are built with a top feed, with valves so arranged that only a part or all of the press may be used at a time.

It is sometimes desirable to filter quantities so small as not to fill all the plates of the press. In this case, a blank, or dummy plate, is used to cut off any portion of the press. This is a solid plate without center channel. To use the dummy plate, it is inserted between any two plates where it is desired to cut off the flow. For example, if only five chambers of the press are wanted for use, the dummy plate is inserted between the fifth and sixth plates, the press screwed tight, and thereby made a perfect working press of five chambers. This plate is very convenient and should accompany every press.

TALLOW, GREASES, AND OILS

TALLOW

5. Tallow is the rendered fat of cattle, calves, and sheep. Goats are occasionally slaughtered in the packing houses in this country, but their number at the present time is of no industrial importance, although their fat may also be rendered into tallow.

The tallows emanating from the packing house include *edible, prime, packer (No. 1, or packer B, tallow), No. 2 tallow*, and, at times, *acidless cylinder tallow* and *cake tallow*. Between the ordinary tallow and oleo stearin comes the tallow stearin, an article having some of the characteristics of both the former products. The manufacture of this stearin is described later.

6. Edible Tallow.—The highest grade of tallow is that known as edible tallow, which, as its name implies, is used in food compounds, such as lard and “cotto” compounds, etc. Edible tallow is made from high-grade beef fats that have been subjected to thorough washing in ice water to remove blood and other impurities before placing them in the tank for rendering. It is again washed here by heated water before the tank is closed for rendering it under pressure. Fats from which edible tallow is made are of a grade suitable for the making of oleo oil and stearin, but are of a troublesome size. The material is carefully rendered in the usual manner, especial care, however, being used not to subject it to too high a temperature or to cook it too long. A pressure of 25 pounds of steam per square inch for 8 hours will give good results in obtaining a high-grade material. As an advanced price over the regular prime tallow is always obtained, the extra preparation and care is thus repaid. This tallow is usually made only when the demand for it is very apparent. The hardness, or titer, of this is not of such moment as the amount of free fatty acid, which must not exceed $\frac{1}{2}$ per cent.

7. Prime Tallow.—Prime tallow is a tallow made from the usual run of fat from the bullock. That is, the material consists of the fat ordinarily selected for the manufacture of oleo oil and oleo stearin, together with the other portions of good, sweet (not putrified) fat. The material is cooked together in the pressure tank, being subjected beforehand to the usual washing in the tank. A pressure of 30 pounds of steam per square inch for 5 hours is the usual method of cooking. The color of this material when drawn into barrels

and chilled must be a clear yellow and not grayish or of any decided shade. If white and clear, it is the more desirable. The free fatty acids in this tallow should not exceed 4 or 5 per cent., and the titer must be at least 42° C.

8. No. 1 Tallow.—The tallow most largely produced in the packing house is the ordinary tallow of trade known as packer B, or No. 1, tallow. This product is made from the regular run of fatty material (other than the material picked for oleo making), consisting of all fats of any description from the bullock, calf, and sheep, which, when rendered, will produce a tallow of good or fairly good yellowish color. The stock for this is subjected to soaking and washing in the tank before rendering, as, in common with all material of this nature, the more washing it receives, the better will be the resulting product. As this fatty material is almost always accompanied by more or less filth, slime, and dirt, vigorous washing is frequently essential to produce an article grading as No. 1 tallow.

The same cooking as that given the prime tallow is also given to this. The lighter the color and the cleaner the tallow, the better is the market price and sale of the commodity. The free fatty acids in this tallow are seldom considered by users, although within very recent times much closer attention is given to their presence in this particular grade of tallow. Ordinarily, if of good color and practically free from moisture and impurities (less than 1½ per cent.), it is accepted at a price dependent to a large extent on its hardness. To be merchantable, this tallow must have a titer of at least 42° C.; and, if higher, a correspondingly higher price is paid. At the present time, besides the test for titer, tallow is subjected to chemical analysis for moisture and impurities, upon the results of which sales are based. The industrial uses of this tallow are too well known to need mention. As the titer of tallows is such an important feature, both for the producer and consumer, a detailed method for its determination will be given further on.

9. No. 2 Tallow.—This grade is made from all tallow yielding material from which the other selections have previously been made and from the pressings of the beef tankage and fertilizer material. It is the final place to which any and all kinds of tallow-yielding stock is consigned. There are no especial requirements for this tallow other than freedom from moisture and impurities. There is no recognized standard, each lot being sold on its respective merits, in respect to the above features and its titer.

The color of No. 2 tallow varies widely—from yellowish green to brownish. The free fatty acids are frequently as high as 20 per cent. or even more, depending on the source of material from which it is made. The tallow derived from livers, lungs, floor scrapings, etc. also constitutes No. 2 grade. It is never bleached and is seldom pressed for oil. The industrial uses of this tallow are principally for distilling into oleic and stearic acids and glycerine. A small amount is consumed by soap makers. When the percentage of free fatty acids is excessive, it cannot be used profitably for the production of glycerine or stearic acid.

The offal from calves is placed with the beef material, yielding a small quantity of No. 1 tallow and a very little No. 2 tallow.

10. Acidless Cylinder Tallow.—This grade is made from tallow of good grade, the fatty acids of which do not exceed 5 or 6 per cent. The operation of making this acidless is carried out in the same way as the making of tallow oil into the acidless condition, which will be described later. This tallow has at the present time a limited sale, its principal use being a lubricant for locomotive and other engine cylinders.

11. Cake Tallow.—This is a good grade of tallow, having a titer of at least 44° C., and is made into the form of cakes, which weigh about 5 pounds each. It is usually packed into boxes holding twenty cakes. The form and shape of the latter are a matter of individual taste. The most common form is a cake about 3 inches

thick and 6 inches square. This tallow is generally white in color, but need not be, if the requirements as to hardness and freedom from impurities are acceptable. The harder the tallow, the better it suits the consumer. This tallow is made by running the warm and molten tallow into sheet-iron molds of the required dimensions and chilling quickly by any convenient means, without allowing the material to grain by separating into oil and stearin. The cakes when thoroughly chilled may be easily knocked from the pans and immediately packed.

The cost of making this tallow is about a quarter of a cent a pound more than tierce tallow, but the selling price is greatly in excess of this extra cost. It is a very profitable article of manufacture. Cake tallow finds use in the lumbering regions, both for lubricating machinery and for lubricating the runways for lumber and logs. In the lumbering industry, owing to its general utility, it has not yet been replaced by oils. When tallow suitable in all respects for cake tallow, except in hardness, is wanted for this purpose, an addition of a few per cent. of tallow stearin will enable it to fill the requirements.

12. Mutton Tallow.—Where large numbers of sheep are slaughtered, the tallow from the offal is cooked by itself and produces a white, hard tallow known as mutton tallow. The usual method is to cook the heads and other material, other than lungs, livers, and feet, in an open vat with live steam for 10 to 12 hours. After a period of settling the resulting tallow is collected, made free from water, and tierced. The offal from the intestines, feet, etc. produces a dark-colored material, which is tanked with the usual material for No. 2 tallow.

The tankage remaining from the open cooking in the vats is again cooked under pressure with the ordinary No. 1 tallow stock, materially contributing, if in any quantity, to raising the titer of that grade. Mutton tallow has a titer of from 44° to 48° C., the latter when made from the caul and kidney fat. This tallow is very serviceable for

the making of cake tallow and for putting in soft lard, when desired to give a firmer body to the latter.

13. Yield of Tallow.—The yield of tallow from fat is very variable, depending very largely on the material from which it is made. When the total fat of a bullock, other than that left with the dressed beef, is tanked, the yield approaches 74 to 78 per cent. of tallow. This includes the caul fat and the other parts generally selected for oleo oil and stearin. This yield can be increased to 80 per cent. by careful and thorough pressing of the cooked tankage after rendering.

When the general run of fat for ordinary tallow is tanked, and the highest grades removed, the yield approximates 68 to 72 per cent. of the material cooked. The amount of tallow obtained in all cases depends on the quality and fatness of the animals slaughtered, together with the care taken in rendering.

The yield of tallow from bones, as with other things, is very variable. When tallow is derived from bones alone, it is known in the packing house as *butter-stock tallow*. It is in reality a bone oil. It is a soft, yellowish-white material that may be, and at times is, utilized for making the lower grades of oleo oil. The tallow from the heads of cattle is the same material as that derived from the other bones. It has a titer of about 42° to 42.5° C.

Kidney fat when tanked by itself may be made to yield from 90 to 95 per cent., the latter in exceptional cases. Caul fat and ruffle fat when cooked under pressure will yield from 78 to 84 per cent., depending on the quality of the cattle from which they are taken.

14. Bleaching of Tallow.—Besides the usual bleaching of tallows with fullers' earth, a mixture of sulphuric and nitric acids in equal proportions is sometimes employed for this purpose. This treatment also hardens the tallow by the chemical action of the nitric acid on the oleic acid, transforming some of the latter into palmitic acid. For 100 pounds of tallow treated for bleaching and hardening,

1 pound each of sulphuric and nitric acids, largely diluted with water, is used, the tallow being washed afterwards to eliminate the excess of acid not used up. Great care is required in this process, which is applicable only with hot stock and in a wood or lead-lined tank.

GREASES

15. A White Grease.—Greases in the packing house are divided into several grades. The highest grade, A white, is made from hog material that would normally make lard, but from various causes is made into white grease. All hogs condemned at slaughter and similar pork material furnish the source of this grease. It is very similar to lard in appearance, odor, and color, and is, in fact, lard except in name. This material furnishes a source of winter-strained lard oil. The stearin from it is not lard stearin, but must be branded white-grease stearin.

16. B White Grease.—Next in grade is B white grease. This is from material not dark or discolored, the resulting grease being of a light fawn or buff color and having a strong, more or less rank, smell. Boiled-out ham and sausage-room grease furnish a plentiful source of this grease, as do also spoiled fresh meats. The lard oil from B white grease is usually light in color, but makes no better grade than No. 1 lard oil.

17. Yellow Grease.—This grease is made from rather dark-colored material or good grade material intermixed with a small quantity of poor material. For example, grease from ham boiling, which by itself would be B white, when mixed with grease from the livers, lungs, etc. becomes yellow grease. In small establishments where all grease material is cooked together, the resultant product is yellow grease. This grease has a strong, rather nauseating, smell, and the color, as the name indicates, is yellowish. Yellow grease yields on pressing No. 1 lard oil and yellow-grease stearin.

18. Brown Grease.—Brown grease is made from all refuse grease-yielding material in the packing house. The pressed-out grease from tankage is placed with this. The dark-colored, greenish-black grease yielded from livers, lungs, and floor scrapings is brown grease. This grease is largely exported to France for distillation into olein, stearin, etc. It is consumed in a small way in the manufacture of cheap soaps, axle grease, etc.

19. Greases are obtained from the fatty material in the same way as lard, the process being carried out in the same manner in every way. Greases of good grade are washed in the tank the same way as lard material, but with the lower grades, such as yellow and brown, no washing is performed. The tankage from grease material is treated the same way as the regular lard tankage.

20. Bleaching of Greases.—Greases are seldom bleached by means of fullers' earth, although, if it can be done economically, there are no objections to the practice. But, as a rule, owing to the time and labor, together with the cost of materials, shrinkage, etc., it is seldom a paying process, the enhanced value of the grease not warranting the expense.

Chemical bleaches are frequently given to greases to make those of dark color more acceptable to the soap maker. With some greases not too black, such a bleach is often an inexpensive and profitable method of making light-colored greases. In the large packing houses, chemical bleaches are not used, the greases being sold in their original condition.

The following method is very serviceable for bleaching light greases and for reclaiming lard that has become old or slightly rancid. For every 500 pounds of stock, $\frac{1}{2}$ pound of potassium carbonate, 2 ounces of sodium acetate, and $\frac{1}{2}$ pound of borax are dissolved in 2 quarts of hot water, and the solution added slowly to the lard or grease, which should be heated to 160° F. The heating is continued while the material is being constantly agitated, by any convenient means.

until a temperature of 200° F. is reached, when the heat is shut off and the material allowed to settle. When cooled to 125° F., the bleached material is drawn off through muslin or a double thickness of cheesecloth to free it from floating scrap, etc.

Another bleach applicable to the same grade of greases is a mixture of 1 pound each of boracic acid and potassium chloride and $\frac{1}{2}$ pound of saltpeter, the whole being well mixed. The material to be bleached is treated with the mixed chemicals in the proportion of 1 ounce to every 30 pounds of grease stock. The procedure is carried out in the same manner as in the preceding method.

21. The following process is extensively used on a large scale to bleach greases. It is a modification of Watts's bleaching process and is employed very successfully; hence, it is given in detail. It is always advisable, as is done in the packing houses with all materials of this nature, to experiment first upon small quantities of material before handling larger quantities. The green color occasioned by the chromic compounds sometimes clings persistently to the grease, but thorough washings will eventually remove it. The method of procedure is as follows:

For 20 barrels of grease dissolve 3 pounds of potassium bichromate, 1 pound of sal soda, and 4 pounds of potassium permanganate in 5 barrels of water. Run this mixed solution into the melted grease, which should be only warm enough to liquefy it. Then thoroughly mix the above solution with the grease by applying a strong current of air. Blow for about 30 minutes and mix 8 quarts of 66° Baumé sulphuric acid with 6 quarts of water, taking care to pour the acid into the water (not reversing the proceeding). Add this diluted acid to the mixture of grease and solution of chemicals, thoroughly mixing the whole by air-currents from an air compressor or blower. When the grease turns green, turn on steam. After 5 minutes have elapsed turn off the air, but leave the steam on and bring the mixture to a lively boil. When boiled for 10 minutes, turn off the steam

and allow the mixture to settle for 10 to 20 minutes; draw off the mixture of water and chemicals and turn on a spray of water with a hose, using clean, hot water. Allow the water to settle and draw it off. (This may necessitate warming the grease again if it has cooled too much.) Meanwhile, $\frac{1}{2}$ pound of sal soda should be dissolved in 50 gallons of water in another tank—the washing tank. The grease bleached in the previous operation is run off into this washing tank and boiled with the sal-soda solution for 2 hours; the steam is then turned off and the water is allowed to settle and is drawn off, leaving a light-colored, almost odorless, grease. This formula is effective for ordinary grades of grease. For very poor grades, the amount of chemicals must be increased, even doubled, without, however, increasing the amount of water.

The most suitable tanks for treating twenty tierces of grease (bleaching as well as washing) are built of 2-inch wood staves. They are 4 feet in diameter at the bottom, 7 feet in diameter at the top, and 10 feet high. For treating very poor greases, when relatively strong solutions of the chemicals are used, the tanks are preferably painted with a good asphaltum paint, which is impervious to acids, etc. The tank must be provided with air-blower pipes, open steam pipe, closed steam coil, and draw-off cocks for water and grease.

Occasionally a grease will retain color and odor in a most obstinate manner, but a grease that will not give a satisfactory result is seldom found if the above directions are closely followed. Some greases can be materially improved by subjecting them finally to a bleach with fullers' earth; usually, however, the grease is acceptable and preferable without the last-named treatment. The contact with copper or brass cocks must be avoided in this process. A large quantity of water and good blowing, or agitation, is necessary to eliminate the smell occasioned by the chemicals. There should always be sufficient acid, so that an excess will be present, or the reaction will not take place. This excess is readily washed out by hot water, the ordinary wooden vats in a packing house being used for this operation.

OILS

22. Oleo Oil.—This is one of the important products of the modern packing house, as it forms a most profitable outlet for a large part of the fat of the bullock, that otherwise would be rendered into ordinary tallow. In the manufacture of oleo oil, scrupulous cleanliness is a most important consideration, as without it no first-class product can be obtained, even though the best possible material be used. While cleanliness is important and necessary everywhere in the packing house and with all products, oleo oil is probably the most sensitive of all to deterioration from uncleanly conditions.

The beef fats used in the manufacture of the highest grades of oleo oils are the *omentum*, or *caul fat*, *ruffle fat* (the apron of fat to which the intestines are attached), and the fat of the *heart*. Other fats may be used, but only those mentioned can be used for the best grade. There are generally two grades of oleo oil made, but when the price of this product is high, a third grade is made. To make the latter, under ordinary conditions, is not a paying investment. For this grade, pieces of beef fat of any sort in good condition may be used.

Oleo oils are always sold by the purchaser's inspection. The requirements are absence of pronounced odor, a clean, bland taste, and a well-defined grainy condition, all being essential for a successful and profitable sale. The absence of any one requirement causes a material difference in price.

23. Preparation of the Fats for the Manufacture of Oleo Oil.—The starting point to obtain good oleo oil is when the fat is removed from the slaughtered animal. The fat should not be allowed to touch the floor of the slaughter house or any unclean receptacle. It is taken from the animal and immediately placed in vats of cold water, the temperature of the latter being kept about 50° F. The fat is allowed to remain here several hours, when it is transferred to another vat of clean water of much lower temperature. After remaining here for some hours, until

the animal heat has been eliminated, the fat is placed in water in which pieces of ice are kept in order that the fat may be thoroughly chilled before being hashed. These successive transfers of the fat require about 10 or 12 hours, it being kept in the iced water until about an hour before wanted for hashing for melting into oil. The raw material should not be kept over 48 hours before making into oil, and it is better if used within 24 hours.

The arrangement of the plant for the manufacture of oleo oil is the same as that shown in Fig. 6, *Packing-House Industries*, Part 1. The operation is conducted quite similarly to that of making neutral lard, but differs in some important details, notably in the higher temperatures employed. The process is as follows:

24. Process of Manufacture of Oleo Oil.—The chilled beef fat is placed in the hasher and disintegrated in the same way as leaf lard. The same apparatus may be and often is employed for the manufacture of both oleo oil and neutral lard. The finely minced fat flows through the spout *s* of the apparatus shown in Fig. 6, *Packing-House Industries*, Part 1, into the melting kettle *D* until the kettle (previously warmed before starting) is filled up to within a foot of the top. The paddles attached to the shaft *c* stir the mixture continuously during the operation. The heat is applied gradually and continuously to the water in the jacket, until, when the kettle has been filled with the minced fat, it registers around 155° to 160° F.

The melting fat is cooked at this temperature for 1½ hours, when the operation is completed. The paddles are raised free from the fat and a quantity of salt is scattered freely over the surface of the melted oil, to cause the scrap and water in it to settle below the oil. The material is allowed to rest for 20 or 30 minutes, when the clear, supernatant oil is carefully siphoned off by means of the attached siphon, the outside leg of which is shown at *e*.

The oil flows through a cloth strainer, to remove floating scrap, to the kettle *F*, where a further separation of fine

scrap and moisture takes place. After remaining here at a temperature of not less than 130° F. for an hour, it is again siphoned by the pipe *g* into the settling kettle *H*. All these kettles are provided with water-jackets, as experience has shown that a dry heat on oleo oil is highly detrimental to its quality.

The oleo oil remains in the final kettle for not less than 3 hours at a temperature not over 130° F. From this it is run into the seeding trucks at a temperature of 130° F. A few degrees from this either way materially alters the character of the stock, this being the term for the seeded, or grainy, mixture of oil and stearin. The warm material in the seeding trucks is placed in a room free from drafts and protected from direct sunlight for 48 hours to have the stock resolve itself into the oily and the hard parts of the original tallow or into olein and stearin. The temperature of the *seeding room* must be steadily maintained at 90° F., and no material variations from this point are permitted.

The scrap and water remaining in the melting kettle *D* is withdrawn through the valve *k* and sent to the tallow-rendering tank, to obtain the tallow not obtained at the mild melting temperature. The kettle is thoroughly cleansed, when it is again ready for another charge.

25. Pressing the Oleo Stock.—After the stock in the seeding trucks has assumed the required condition for pressing, it is taken to the pressing room. The oil and stearin by this time are distinctly separated, yet intimately mixed. The material is thoroughly and uniformly mixed with the hands and placed in cloths laid over a mold and wrapped into the form of a cake. To facilitate working, these molds are made on a circular revolving table, so that, with one man filling cloths, one folding into cakes, and another placing them in the press, the work proceeds very rapidly.

26. The form of press used for this work is illustrated in Fig. 3, the iron press plates not being shown. This press is known as the **knuckle-joint press**, and is run by power. The speed of the press is regulated by pulleys, for a rapid

or slow descent, the latter being applied when the material is being pressed.

27. The cloths for pressing the stock are of heavy, closely woven duck, heavy enough to withstand the high pressure applied to them. This point is one of many where close attention to small details is necessary to produce a



FIG. 8

high-grade article, and neglect of this alone will be the cause of an inferior product where all other parts of the manufacture have been properly carried out. The press cloths must be thoroughly washed after each day's run and dried free of all moisture. While cloths for pressing other oils may be used several times, those for pressing oleo oil are used only one day without washing and drying.

The material, folded in the cloths into the form of cakes, is placed in the press on the bottom fixed plate until the latter is covered with a sufficient number of bags. A sheet-iron plate is then inserted in the press and dropped on the bags. This plate in turn is filled with material and another plate inserted above, this performance being repeated until the press is filled to its capacity, which varies with the size of the press used. The oily part of the stock begins running out long before the press is filled, owing to the weight of the upper plates and material pressing on the lower layers. This is oleo oil, or, as it is termed in Europe, *oleomargarine*. This term must not be confounded with the American name oleomargarine, which name applies exclusively to factitious butter, or butterine.

The oil flows to the settling receptacle, which should best be water-jacketed, the flow of the oil being augmented by the ram of the press slowly descending and pressing the material. The speed of the press while pressing is very slow, occasional periods of rest from descending being allowed in order to give the oil time to ooze from the stearin. The operation of pressing is completed in about 30 minutes, but the stearin in the bags has the pressure left on it an hour longer to give the material ample time for draining. When the stearin is free from oil, the ram of the press is rapidly raised and the press unloaded, the operation being exactly the reverse of filling. The oleo stearin is removed in the cloths, and the press is then ready for another charge. The temperature of the press room should always be around 90° F.

The oleo oil from the pressing is held for a short settling period in the reservoir, from which, at a temperature of 92° F., it is drawn into tierces, which are immediately placed in a temperature of 55° to 60° F., to acquire the requisite grainy condition. The temperature must be kept within these limits to insure successful graining, which takes place in from 5 to 6 days. When placed in the graining temperature, the 3-inch side bungs of the tierces are removed, to allow heat and any possible odor to escape. Oleo oil finds

exclusive use in the manufacture of oleomargarine, or butterine. The greater part of this oil is exported to Holland, from which point it is distributed throughout the various European markets.

Oleo oil should have a titer not exceeding 40° C.; but at times this may be exceeded without detriment, as the oil is not purchased on its titer test, but on its physical characteristics only, as noted. As the raw fat from which this material is made is kept in a cold, fresh condition, free fatty acids cannot be generated, and, consequently, the free fatty-acid tests of oleo oil will show ordinarily less than $\frac{1}{2}$ per cent. Good oleo oil contains but .2 or .3 per cent. free fatty acids. The yield of oleo stock from fat is about 70 per cent., and the yield from pressing the grained stock is about 50 per cent. each of oleo oil and oleo stearin. This percentage yield is governed largely by market conditions, the manufacturer inclining to the greatest yield in the highest-priced product, whether oil or stearin.

28. Oleo Stearin.—After the oleo oil is pressed out as just described, the oleo stearin is shaken free from the cloths into a bin, and from this it is filled into large, thin-staved tierces, which weigh when filled approximately 600 pounds. The oleo stearin is pounded as compact as possible into these tierces, the object being both to have as little air as possible intermixed with the material and also to have them hold as much material as can be pounded in with the large wooden maul. For export purposes the stearin is sometimes melted and run into tierces, the material in this condition retaining its sound condition for a very long period. Oleo stearin finds a very large outlet in the tanning and leather trades, and also for soap and candle purposes. It is quite largely used in the manufacture of lard compound and, at times, in lard.

Oleo stearin is pressed so it will have a titer of from 48° to 52° C.—generally averaging about 50° C. The harder the oleo stock is pressed, the harder the stearin will be until the limit is reached. This is about 52° to 53° C.; but

oleo stearin of this titer is never made commercially. To pass sale requirements, the titer must be at least 48° C.

The apparatus required for making oleo oil and stearin comprise the kettles illustrated in Fig. 6, *Packing-House Industries*, Part 1, and an oleo press, as shown in Fig. 3, together with the necessary seeding trucks, cloths, wringer for the latter, skimmers, etc. The complete outfit may be purchased at a cost of about \$1,200, not including boilers, power, or building.

29. Neatsfoot Oil.—The method of obtaining neatsfoot oil has already been described. This is known in the trade as *pure neatsfoot oil*, *extra-prime neatsfoot oil*, etc.

Another grade of this oil is made from the refuse of the first boiling. This material is technically termed *jelly*. With this is sometimes placed the grease caught by the catch basins in the beef department of the packing house. Care must be taken, however, not to have too much tallow in this oil, as it will remain solid in the barrel. The jelly and grease are boiled with live steam in open vats for 24 hours. A large excess of water must be added to this material to allow the freed oil to rise through the glue liquor formed by the operation. The resultant oil is more or less dark colored, has a strong smell, and contains, as a rule, a high percentage of free fatty acids. This oil is known as *No. 1 neatsfoot oil*.

30. Winter-Pressed Neatsfoot Oil.—To press neatsfoot oil into winter-pressed oil and neatsfoot stearin, it is necessary to have the oil in a grainy condition. By this is meant a separation of the olein from the stearin—the solid portion of the oil. The pure neatsfoot oil, in barrels, is placed in a temperature of about 32° F. for 4 weeks. Various unsuccessful methods have been tried to shorten this time. It is usual to set the barrels on end, with one head removed.

After the oil has become in condition for pressing, it is placed in cloths of closely woven duck. The bags when folded contain about 5 pounds of material. In pressing this oil, a very essential point is to use a double cloth for

enfolding the material or an oil of high cold test will not be obtained. The temperature of the press room should be kept steady, at 28° to 30° F., when the pressed oil, using double cloths, will have a cold test as low as 14° to 16° F. But the manufacturers will seldom guarantee winter-pressed neatsfoot oil to withstand a cold test under 20° F., owing to the variable and uncertain methods of making the cold test by purchasers.

31. Pressing Neatsfoot Oil. — The details of pressing are as follows: The cloths are placed on an upright wooden mold made so that when folded the cloths form a bag about 9 in. \times 6 in. \times 1½ in. These bags are placed on the bottom of the press, shown in Fig. 4, with room between them to allow for the spread in pressing. A plate of sheet iron is then placed on these, thus forming another bottom for making the second tier of bags. This operation is repeated until the press is filled with layers of bags alternating with the separating plates of iron. The top plate *b* is usually of wood strong enough to withstand the pressure without bending, thus keeping a flat surface and an equal pressure on the top layer. The chains attached to the ratchet *h* are then connected to the bar *c* on both sides by means of iron links, the chain, not shown in the figure, being fastened to the axle corresponding to *h* on the other side of the press.

The slack in the chains is taken up by raising and lowering the lever *k* until the lever remains raised in the air. The framework *a, a* of the press is made of strong angle iron, in which the plates fit easily at the corners, so that they can follow down the pressed bags of oil. The lever *k*, at first without weights, is raised as quick as it falls to the floor. As soon as the resistance offered by the material in the cloths keeps it from falling, a cast-iron weight *m* is placed on the notch in the lever and the weighted lever again raised as fast as it falls to the floor. When it remains suspended with one weight, another weight *m'* is added to the lever, and the same performance is repeated until, with

both weights on, the lever remains in the air. The material in the cloths during this time has been constantly deprived of the liquid portion that flows to the reservoir through *g* from the pan *f*. This oil is neatsfoot oil of low cold test, not congealing much above 14° F. The cold test of the

FIG. 4

oil above this point may be regulated by the temperature of the press room, but it is neither practicable nor profitable to produce oil withstanding a lower cold test than that above mentioned.

The time required to complete the pressing is about 3 days, the pressing proceeding very slowly after the bulk

of the oil has oozed out. The levers must be frequently raised the first day, and thereafter about twice every 24 hours.

32. Neatsfoot Stearin.—The material remaining after pressing is actually neatsfoot stearin, but it is seldom sold as such. The close approach in its nature, composition, and cold test to tallow oil makes its sale as that commodity legitimate. In warm weather, neatsfoot stearin may be sold for neatsfoot oil, as the cold test at this season is not important. The bags or cloths are shaken free of the pressed material, when they are ready for use again without washing. Cloths for pressing neatsfoot oil may be used four or five times without washing, but the texture is gradually closed up by waxy-like stearin, which necessitates removal. The neatsfoot stearin is simply melted and either barreled for neatsfoot oil or used and sold for tallow oil.

Winter-pressed neatsfoot oil is largely employed for a lubricating oil on very fine instruments, as watches, chronometers, etc. The pressed oil is usually heated to eliminate accumulated moisture and filtered to remove extraneous contaminations and any accidental dirt. The pressed oil may be, but seldom is, bleached white by means of fullers' earth. The sale for this class of oil is very limited. The process of bleaching is in every way similar to the bleaching of lard, which has already been described.

33. Lard Oils.—Lard oils are made from lard and grease, the same kind of press (Fig. 4) being used for this as for pressing neatsfoot oil. In large establishments, several of these presses are arranged side by side, the pan *f* being made sufficiently long to accommodate all the presses. One outlet *g* serves to drain the oil from all the presses. The axles of the presses are placed at such an angle that the levers clear themselves in falling. The advantage of having several presses in this way is that while one press is having the material pressed, another may be filled. As it requires 2 to 3 days to press oil out of lard and grease, the advantage of such an arrangement is obvious.

34. Pressing of Lard Oils.—The same method is used for pressing lard and greases, much greater care being observed, however, in handling the lard, both before and after pressing, than is necessary with greases or the ordinary lard oils.

The lard is made into a grainy condition suitable for pressing. The more marked and complete the separation of the olein and stearin of the lard, the better and easier it can be pressed. Lard or grease that has not been made into a grainy condition is not susceptible of pressing, as the oil and stearin, being intimately mixed and in a smooth, plastic condition, resist separation by pressure. To obtain the material in the requisite form, the lard or grease after being filled into the receptacles in a molten condition is allowed to remain quiet for 3 or 4 days in a temperature of about 60° F., when the separation of the material takes place. A longer period of graining will do no harm, and at times is a positive advantage, as under favorable conditions a most complete separation of the oil and stearin ensues, enabling the lard oil to be dipped from the stearin almost completely. Many small concerns are thus in a position to obtain oil and stearin without the use of a press. This is, however, not a general way of obtaining the lard oils.

The grainy lard or grease is filled into cloths similar to the manner of pressing neatsfoot oil and placed in the press layer on layer until the latter is filled as high with material and plates as practicable. The process is similar to that of pressing neatsfoot oil. With lard and grease, a single cloth is sufficient to permit the lard oils to flow through and at the same time retain all the stearin in the cloths.

35. Winter-Strained Lard Oil.—In pressing prime steam lard, the material from which winter-strained lard oil is made, the most scrupulous cleanliness is observed, as the resultant lard stearin is sold on the same characteristics as the lard itself. That is, it must have a sweet, mild smell, have a clear, natural whiteness, and be free from all objectionable features. The merchantable titer of lard stearin

must be at least 44° C. This necessitates pressing in a temperature of 55° F. and the temperature should not vary more than two or three degrees either way. Prime steam lard must be in the press generally about 2 to 3 days, the levers being raised and weights attached thereto, as occasion demands. Winter-strained lard oil, to pass requirements, should not contain over 2 per cent. of free fatty acids and should possess a mild, sweet smell.

36. Extra No. 1 Lard Oil.—This is a grade intermediate between winter-strained and No. 1 lard oil. While not answering the requirements of the former, it is in every way superior to the latter. Extra No. 1 lard oil is made from material not suitable for the best grade lard oil and much too good for poor grades. Pig's-foot lard, old or unmerchantable, steam, or kettle lard, lard from condemned slaughtered or dead hogs, etc. furnish material for this oil. In color it is generally whitish or very light yellow and contains a variable percentage of free fatty acids, not, however, exceeding 5 or 6 per cent. It is usually of mild odor. If this oil is rather dark in color, it is customary to bleach it as light as possible, by the usual method, with fullers' earth.

37. No. 1 Lard Oil.—This is the next lower grade of lard oil. It is made from light-colored grease and is ordinarily bleached as light as possible, by means of fullers' earth; the lighter the oil, the better the price obtained. Although light-colored grease is preferable for this oil, any material of this nature susceptible of after bleach may be used. A very small amount of dark, unbleachable grease will discolor many times its own volume of light-colored grease. From this it will be seen that it is impracticable to raise the grade of dark-colored oils or any other fatty material by mixing with them light-colored material of the same kind. No. 1 lard oil has a more or less rank odor. The free fatty acids should not exceed 15 per cent. This latter quality is very variable, and other things being equal, the preference in sales is given to oil containing the least amount of free fatty acids.

This grade of oil is very largely made and is used almost exclusively for lubricating machinery and for mixing with mineral lubricating oils. The requirements as to cold test, etc. will be found in the annexed rules governing the transactions in animal oils in the New York Produce Exchange.

38. No. 2 Lard Oil.—This grade has of late years fallen into much disfavor, owing to its replacement by the less expensive mineral oils. It is not manufactured largely at the present time on account of its very limited sale. No. 2 lard oil is made from brown grease—the refuse grease of the packing house. The pressing is carried out in the usual manner, not much care or attention, however, being bestowed on the cold test. The color of this oil is usually a dark-reddish black, but there are no requirements asked or expected in reference to color or free fatty acids. The latter may run as high as 30 per cent. As a rule, the free fatty acids range from 18 to 24 per cent.

39. Bleaching Requirements of the Several Grades of Lard Oils.—All the oils after pressing are allowed to settle in the receiving tanks until a sufficiently large quantity has accumulated to filter and, if necessary, bleach. The oil is placed in the bleaching tank, the air blower started, and the oil heated and agitated until the temperature reaches 160° F., when the necessary quantity of fullers' earth is added and the hot oil and suspended clay are pumped through the filter press. The bleached and filtered oil is sent to the storage tanks, from which, after the oil has been allowed to cool to normal temperature, it is drawn into the regulation oil barrels. It is necessary to allow hot oil to cool before barreling, in order to avoid the oil acquiring a cloudiness in the barrels while cooling. Such oil is technically termed *off*.

Winter-strained lard oil is never bleached, but simply heated and blown, to eliminate the moisture present, and passed through the filter press to remove extraneous impurities.

Extra No. 1 lard oil, if not sufficiently whitish in color, is treated with the required amount of clay, to produce the light color, and passed through the filter press.

No. 1 lard oil may or may not require bleaching, this depending on the grade of material used in its pressing. For some trade, however, it is always bleached, the lightest possible color being desired.

No. 2 lard oil is never bleached; it is simply settled to clarify, and heated to expel moisture. This oil is seldom filtered through the press, clarification by subsidence generally being sufficient.

40. Lard and Grease Stearins.—After the oil has been pressed from the material, the stearin remaining is shaken free from the press cloths, and when a sufficient quantity of it has accumulated, it is melted, mixed to insure uniformity, and run into tierces.

Lard stearin is used to stiffen refined and mixed lards, thereby causing such lards to withstand warm weather without melting. At times, though very seldom, it is used in lard compound.

Grease stearins are divided into several grades corresponding to the greases from which they are made. That from A white grease is branded *white-grease stearin* and is used by soap manufacturers. The stearins from B white and yellow grease are both classed as *yellow-grease stearin*, no distinction being made between them. This stearin is used almost exclusively in soap manufacture, having a titer somewhat higher than the usual trade tallow. Brown grease is seldom met with in trade, it usually, and that very rarely, being made on order.

The requirements of stearins are limited to the titer, or hardness, and the amount of moisture and impurities they contain. The titer required on lard stearin must be at least 44° C., and it must not contain over 1 per cent. of moisture nor any impurities. White-grease stearin, as with yellow-grease stearin, is sold at a price corresponding to its hardness and contained moisture and impurities. They

are usually pressed to a hardness of 43° or 44° C., and contain, as a rule, less than 2 per cent. of moisture and impurities combined. The rapid methods for determining these, practiced in packing-house laboratories and at the present day by many tallow and grease brokers, are given later.

41. Tallow Oil.—Tallow oil is made similarly to lard oils, with modifications as to temperature in pressing. Like lard and grease, the tallow must be in a *grainy* condition before it can be successfully pressed, and, similarly, the better the separation of the olein from the stearin in the *seeding* receptacles, the quicker and better the separation of the oil in the press.

The molten tallow for pressing is drawn into suitable receptacles, such as open-headed barrels or trucks, and allowed to stand for 5 days at a temperature of from 75° to 85° F. The tallow by this time will have resolved itself into the component parts of oil and stearin, and is ready for pressing. It is unwise to allow the grained tallow to remain without pressing longer than this period, as the stearin grains will constantly increase in size, entangling the olein, in consequence of which it is very difficult to press out from it the required yield of oil.

42. Pressing and Bleaching Tallow Oil.—The grained tallow is manipulated in the same manner as grease, and when made ready in the cloths for pressing, it is filled into the molds in the way already described. The cakes are placed in the press, Fig. 4, and the operation completed in the usual way. The tallow is allowed to remain pressing for 2 to 3 days at a temperature of about 85° to 90° F. The higher the temperature of the press room, the greater will be the yield of tallow oil; but, on the other hand, the cold test of the oil will be quite high, owing to the greater quantity of stearin melted into the oil. This procedure of pressing at a high temperature is practiced at times when the price of the oil is higher than that of the tallow stearin. The usual

yield, however, is from 45 to 50 per cent. of oil from the tallow.

The oil is conducted to a reservoir until a sufficient amount has collected for filtering and, if necessary, for bleaching. Tallow oil is ordinarily of a very light yellow or white color. If the pressed-out oil should not be satisfactory in color, it is bleached in the usual manner, about 3 to 6 per cent. of fullers' earth, depending on the stock, being employed. The temperature at which the material is bleached is not of particular consequence, as this oil is not used for food purposes. A good temperature is around 140° F. The oil need not be bleached to a water-white color, as this is not an essential. This oil is used in compounding with mineral lubricating oils.

43. Acidless Tallow Oil.—The first operation in obtaining this grade of tallow oil is to obtain the ordinary tallow oil as described above. In selecting tallow for this purpose, it should not exceed 4 or 5 per cent. of free fatty acids, as, when pressed, these free fatty acids are practically doubled in their relative percentages in the pressed oil, the material left in the cloths—tallow stearin—retaining a very small part of them. The less free acid a tallow contains, the less shrinkage there will be in the operation of making into the acidless condition. The operation is carried out in the following manner.

The oil is brought into the treating tank, which is similar to the bleaching tank shown at *A*, Fig. 4, *Packing-House Industries*, Part 1, and may be used as such, the suitable connections being provided. This tank must have the cock *m* about 4 or 5 inches wide, in order that the soap formed may be withdrawn through it. The other attachments of this tank are the same as for the regular bleaching tank. The oil is brought to a temperature of from 125° to 135° F., and the percentage of free fatty acids having previously been ascertained, sufficient solution of caustic soda is added to combine with the free fatty acids present. The caustic solution is made with a strength of 20° Baumé, as this

strength has been found best for this purpose. During the addition of the alkali the oil is constantly agitated with the air blower.

In a few minutes the reaction is complete, when the oil is allowed to stand at rest for 5 minutes to allow the soap formed by the combination of the alkali and the fatty acids to collect on the top of the oil. A portion of the soap will sink to the bottom with the excess of water. The floating soap is removed by skimming, and the soap in the bottom of the tank is removed, together with the water, through the cock *m*. The oil is then agitated to cause the remaining soap to gather together. This is also removed in the same manner, and the oil is now washed with clean water and agitated strongly with the air blower. The washings serve to remove the last traces of soap and any excess of alkali present. The wash water is drawn off and the oil heated to 165° or 170° F. and blown until the last traces of moisture have been removed. The oil is now ready for bleaching and filtering, which is carried out in the usual manner. Acidless tallow oil is always bleached after being made acidless, and, as before mentioned, the same tank may be made to serve for the complete operation.

The chief trade requirement for acidless tallow oil is that it shall not contain more than $\frac{1}{2}$ per cent. of free fatty acid. The cold test of this oil is of no material importance, although it is generally made to stand a test of about 45° F. The whiter the color, the more acceptable the oil, other conditions being equal.

44. Acidless tallow oil finds its greatest use in compounding with mineral lubricating oils, where the absence of all acidity is of the highest importance. It is also used alone as a lubricating agent for machinery. This oil, as made in the large packing houses, seldom contains over .2 per cent. of free fatty acids. The soap formed in the manufacture of acidless tallow oil, when a sufficiently large quantity has accumulated, is decomposed with sulphuric acid of 66° Baumé in a lead-lined tank. The resulting

fatty acids, black in color, may be mixed, if desired, in small quantities at a time with No. 2 tallow.

45. Tallow Stearin.—The material remaining in the cloths in the press after the oil has been pressed out of the tallow is tallow stearin, corresponding to the grease stearin from the operation of pressing grease. The tallow stearin is shaken free from the cloths, melted, and run into the barrels or tierces while warm. The hardness of this material depends on the quality of the stock from which it is derived and also on the amount of pressure to which the tallow has been subjected. In general, it will have a titer of 46° or 47° C. This material, like tallow, is purchased on the result of the titer test. Tallow stearin finds utility in the currying and finishing of leather, in the candle industry, and at times is used in the manufacture of high-grade soaps.

46. Bleached Tallow Stearin.—This is simply the foregoing product bleached with the required amount of fullers' earth to a white color. The bleaching is done in the usual manner, no notable quantity of fullers' earth being required to produce the desired white bleach. The industrial uses of this product are the same as for the ordinary tallow stearin. Tallow stearin may also be used in the manufacture of compound lard, when the price and quality warrant.

47. Bleaching of Oils.—The bleaching of oils is carried out in the same manner as that of lard. With lard and tallow oils, however, the process need not be so carefully conducted as to temperature, etc.

The temperature required for obtaining the requisite bleach in oils is higher than that required for lard and tallows. But here, also, the lower the degree of heat at which the oils may be made to bleach, the better will be the odor of the product. As these oils are not for food purposes, the taste and odor are not of so much importance as with lard, and the use of an excess of fullers' earth in bleaching is not so closely observed except as a matter of cost.

The quantity of fullers' earth necessary to bleach a given oil varies with the nature of the material. While one oil

will bleach with 2 or 3 per cent. of fullers' earth, another of similar kind may require 7 or 8 per cent. When more than the latter quantity is necessary, as shown by preliminary tests, the attempt to bleach is not made, as the enhanced value of the bleached oil would not equal the cost of the labor and fullers' earth used in the operation.

48. Bleaching Test.—The oils may be tested for the amount of fullers' earth necessary to bleach by taking an ordinary 4-ounce oil-sample bottle and weighing in 100 grams of the oil under consideration. The percentage of fullers' earth judged necessary to effect the bleach is weighed off and added to the hot oil in the bottle. By placing the oil and bottle in a steam bath for several minutes previous to adding the fullers' earth, a temperature closely approximating that obtained in the bleaching tank is obtained. If necessary, the temperature of the oil, which must be at least 170° F., may be tested by a thermometer.

After adding the fullers' earth, the bottle is closed and violently shaken for 3 or 4 minutes to effect the bleach. The mixture is then poured on a filter paper, placed in a hot funnel, and the color of the filtered oil observed. If not satisfactory, another test is made, using a larger quantity of fullers' earth. In this way the amount necessary for the quantity of oil can be known before starting to bleach. With this test, however, it must be remembered that it is crude and but comparative, as the conditions are not the same in the bottle as in the bleaching tank with the air-blower agitation. An oil will always be bleached in the tank with approximately from 1 to 1½ per cent. less fullers' earth, owing to the better agitation and more intimate and continuous contact between the fullers' earth and the oil.

49. In bleaching oils, the same precaution as with lard must be observed, that is, to have all moisture eliminated before adding fullers' earth, or no bleach will be obtained. After the bleached oil runs from the filter press in a bright, sparkling condition, unlike lard, it is run to a cooler or tank,

where it is allowed to cool to room temperature before being drawn into barrels.

The apparatus shown in Fig. 4, *Packing-House Industries*, Part 1, is used in the bleaching of oils. After low-grade oils have been through the apparatus, it is necessary to thoroughly clean everything with which it has come in contact, to remove all traces of free acid and smell.

50. Tests on Pressing Oils.—The results of a test on pressing extra-prime or pure neatsfoot oil are given below. The oil was pressed into winter-pressed neatsfoot oil. The test shows how the cost of transforming one product into another is figured in the packing house, every detail being taken into consideration. The prices given were those prevailing at the time. It is always customary to figure the value of oils *loose*, that is, not in barrels, at 2 cents per gallon less than when sold in these packages, this difference in price being ordinarily the cost of a 50-gallon oil barrel. The tests are based on 100 pounds of material.

100 pounds extra prime neatsfoot oil yielded:

Neatsfoot stearin.....	54.5 lb.
Winter-pressed neatsfoot oil.....	44.5 lb.
Loss.....	1.0 lb.
Total.....	100.0 lb.

100 pounds extra prime neatsfoot oil, loose, cost

(sale value)	\$7.06
Labor used, per 100 pounds of material20
	<hr/>
	\$7.26
Value of 54½ pounds neatsfoot stearin at 6 cents. .	3.27
	<hr/>
Net cost of 44½ pounds winter-pressed neatsfoot oil.....	\$3.99

1 pound oil costs.....	\$.0896
1 gallon pressed oil (7½ pounds) costs,	
loose.....	.6720
1 gallon pressed oil (7½ pounds) costs,	
in barrel.....	.6920

Thus, the actual cost to the manufacturer of making 1 gallon of winter-pressed, pure neatsfoot oil is in the above case, at the prices given, 69.2 cents.

A test on pressing tallow into oil and tallow stearin gave the following results:

100 pounds of tallow, at a labor cost of 36 cents per hundred pounds for all operations, yielded:

Tallow oil.....	50 lb.
Tallow stearin.....	49 lb.
Loss.....	1 lb.
Total.....	100 lb.

Butter-stock tallow when pressed yields a much larger quantity of tallow oil, owing to the small amount of stearin present in comparison with the olein. A test made on this material gave for

100 pounds butter-stock tallow:

Tallow oil.....	72 lb.
Tallow stearin.....	27 lb.
Loss.....	1 lb.
Total.....	100 lb.

In figuring oil-pressing tests in the packing house, it is always customary, even if the actual weights show that no losses have occurred, to estimate pressing losses as 1 per cent. This loss may at times be exceeded. If this is the case, the actual loss must be figured in to obtain the true results.

51. Rules Regulating Transactions in Oil.—The following are excerpts from the rules regulating transactions in oil (other than petroleum and cottonseed oil) among members of the New York Produce Exchange:

RULE 3.—All lard oil shall be tested by the following method: The oil to be tested shall be taken from at least one-quarter of the barrels sold or delivered, and when well mixed together, shall be placed in a glass bottle such as is now used to test the specific gravity of paraffin and other

oils, and shall not be over 4 or under 2 inches in diameter, nor over 10 or under 6 inches in height. The thermometer shall be placed in the oil, and when it indicates the degree to be tested at, it must not be allowed to go more than 1° below or 1° above that point. After remaining 4 hours, if the oil shows no evidence of congealing, it shall constitute a good delivery.

RULE 4.—Tares shall be tested, if required by either buyer or seller, by emptying 5 barrels in each 100 barrels to be taken indiscriminately from the lot.

ANIMAL OILS

RULE 5.—Prime lard oil shall be bright and sweet, and shall have the flavor of good sound lard. Whiteness with lack of flavor, or any indication of rancidity, shall not constitute prime or contract oil. All animal oils shall be sold by weight, at the rate of seven and one-half ($7\frac{1}{2}$) pounds net to the gallon.

RULE 6.—The winter test of lard oils of all grades shall be 43° F. or under. The spring and fall test of lard oils of all grades shall be 55° F. or under. The summer test of lard oils of all grades shall be 65° F. or under.

RULE 7.—All contract oil must be in good, sound, iron-bound barrels, holding not less than 42 or more than 50 gallons. In settling contracts without delivery, the basis shall be 46 gallons per barrel.

BEEF EXTRACT

52. General Remarks.—In the making of beef extract in the packing house, the product of beef only is used. It is, with the necessary apparatus, a very profitable product, as in most cases waste liquors, washings, and waters from meat boiling are utilized for this purpose, which are practically without value for other purposes.

The manufacture of meat extract as carried on in packing houses and the meat-canning establishments of this

country constitutes merely a side issue, and comprises essentially only the concentration of suitable meat juices to a proper consistency. Only on rare occasions is fresh meat used, especially for meat-extract purposes, and even then it is only head meat or cheek meat, hearts, and other cheap meats, and these only when *sausage meat* is a drug on the market.

For a short period, some packers who make sausages on a large scale thought it profitable to thoroughly soak all their sausage meat, press the juice from the meat, and use this juice for meat extract. While they found this profitable for the extract department, it was greatly to the detriment of the sausage department. Sausages made from such extracted meat have a very flat taste and no keeping qualities.

The system of making meat extract by soaking the fresh meat with a relatively small amount of water, and pressing the juice from the meat with strong hydraulic presses, is operated in this country only by one small concern to a very limited extent. If prepared with the greatest care, a great deal of skill and experience, and in the very best obtainable vacuum (of about 29 inches barometric pressure, at a temperature below 120° F.), requiring a special and expensive vacuum pan, this meat-juice extract represents a very superior article and brings a high price, but the market is very limited, and the one small factory in the United States finds difficult sale for its product. This process is too expensive to be employed in the ordinary meat-canning or packing establishments.

The waters in which fresh meats have been soaked (the so-called *cellar waters*), together with the waters from the cooking or scalding of meats for canning and certain bone liquors, hereafter described, constitute the main sources of beef extract. The entire manufacture from such liquors is quite simple and relatively inexpensive, the cost being practically the labor and steam employed. Figs. 5 and 6 illustrate the apparatus used in the manufacture and show the principle of the operations.

53. Process of Manufacture of Beef Extract.—The process for manufacturing beef extract from meat liquors, etc. is carried out in the following manner:

The scalding liquors (soup waters) from the cook room of the canning department, bone liquors, etc. are pumped into the wooden vat *A*, Fig. 5, which is about 2 feet high.

FIG. 5

and of suitable length and width to hold 1 day's soup liquors, etc. Instead of using one very large tank, it is advisable to use two or three smaller tanks. The tank or tanks are provided with 1-inch steam pipes, sufficiently far apart so that the pipes and the bottom of the vat beneath the pipes can be readily cleaned. The tanks are slightly inclined towards one corner, to facilitate complete draining and washing out.

The soup liquors and bone liquors are boiled down in tank *A* to about one-half or one-third their original volume. Then an equal volume (or thereabouts) of cellar water (from the soaking of fresh meat) is pumped into the soup water. By continued heating of the mixture, the albumin in the cellar water is coagulated, and while coagulating envelops all the suspended impurities. Steam is turned off and the liquor allowed to settle. The amber-colored clear liquor is drawn from vat *A* through cock *c* and the hair sieve *d* into the vat *B*, which is arranged similar to vat *A*. In vat *B*, the liquor is further concentrated to a density of about

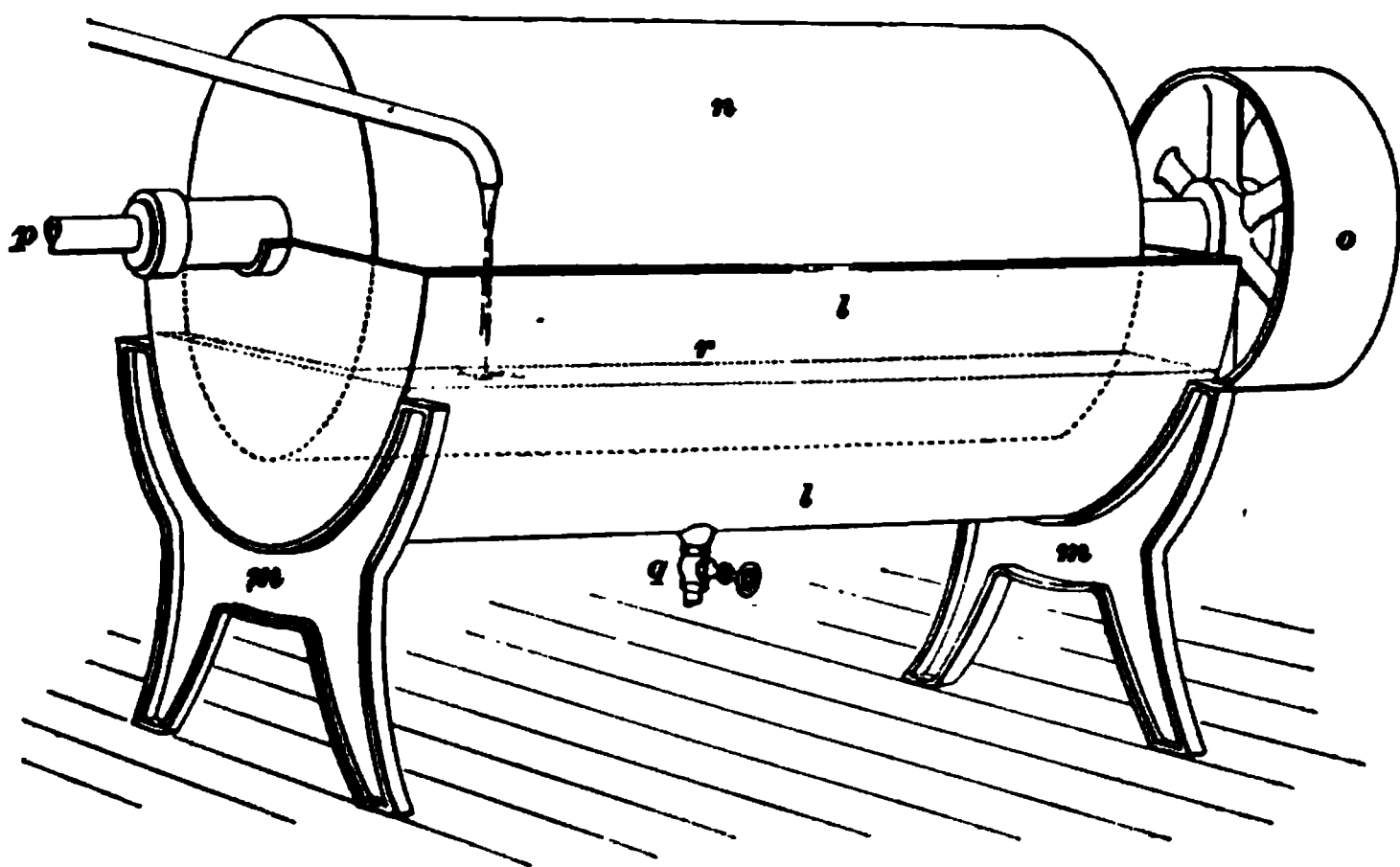


FIG. 6

7° to 8° Baumé. If the liquor shows any tendency to become cloudy or off-colored, a little more cellar water is added to the liquor, which produces a second coagulation and clarification of the liquor. Steam is then shut off on vat *B* and the liquor is drawn through cock *e* and the valves *f* into filter bags *g*. These bags, made of cotton flannel (about 15 inches square on top and 2 feet deep), are suspended by the hangers *h* from a suitable rack. The gutter *j* catches the clear, filtered liquor and conveys it to the rotary evaporator. This evaporator, shown in Fig. 6,

consists of a steam-heated, revolving drum *n* driven by the belt on the pulley *o*. It is about 3 feet in diameter and 5 to 10 feet long, revolving in a trough *l*, supported by the legs *m*. At the back of the steam-heated roller *n* and at its side, scrapers are arranged to scrape the surface clean. The entire roller is furthermore supported in a frame, which permits the raising and lowering of the roller according to the amount of liquor present.

The amount of liquor in the trough *l* is so regulated that the drum *n* dips into it only to about the depth shown by the line *r*. The steam drum can withstand boiler pressure, but it is usually run with not more than 20 pounds pressure per square inch, and frequently with less. In its revolutions (about 25 to 40 per minute), the drum carries sufficient liquor on its surface to prevent the complete drying and burning. When the desired density is reached, which is about 20° Baumé for the fluid extract and 32° Baumé for the so-called solid or paste extract, the concentrated liquor is drawn through cock *q* or dipped over the edge of trough *l* into suitable receptacles, from which it is filtered into bottles or jars. Such is the principle of the most simple and inexpensive meat-extract apparatus in operation, and furthermore it is a very convenient one.

The larger packing houses and many others have, however, abandoned the use of these roller evaporators, simply because better evaporators are offered in the different multiple-effect evaporators, but these are only suited for operations on a large scale, when from 10,000 to 50,000 gallons of meat liquors are concentrated in 24 hours. For a small concern, the labor and attention required by these evaporators are too expensive, and overcome their principal advantage—the saving in fuel. A rotary evaporator will evaporate, for each pound of steam delivered to the apparatus, about 1½ to 1¾ pounds of water, thus getting an evaporation of about 10 pounds of water from each pound of coal. The average double-effect evaporators give 16 pounds of water evaporation for each pound of coal of the same class; the triple-effect evaporators give 24 pounds evaporation.

The rotary evaporator is very suitable for the concentration of tank liquors or tank waters on a small scale, and is used for this purpose by some packers. A few details need to be considered in the manufacture of meat extract.

54. Soup Liquors and Rib Bones.—All the cooking and scalding waters of fresh meats and of mildly cured salt meats can be used, and mixed with them is a small quantity of corned-beef soup water. The scalding water of strongly pickled meats used alone give an extract too salty, and of a strong, pungent odor and taste, due to the saltpeter contained in it. All the cellar waters ("soak" waters from fresh meat) can be used, even if they are slightly tainted; heating and coagulating these liquors removes all objectionable odor, provided it is not too strongly developed by advanced decomposition.

All canning bones, i. é., bones from meats cut for canning, thigh bones, etc., give, when heated for an hour or two with lukewarm water (about 125° F.), a bone liquor, which is extremely well adapted as an addition to the soup liquors in the manufacture of meat extracts. Care must be taken, however, not to use the water too long or too hot on the bones, as otherwise the liquor dissolves too much gelatine and gives to the meat extract a gluey flavor. For the same reason, bone liquors alone do not make meat extract, but when blended with soup liquors and cellar waters, an excellent beef extract results.

Where large quantities of cheap meat are available for beef-extract purposes, such, for example, as hearts, cheek meat, etc., the meat is cut into small pieces, about 1-inch cubes or thereabouts. This meat is then soaked in ice-cold water for 24 hours, with frequent stirring up of the mass with forks. After 24 hours, the first water is drawn off and pumped to tank *A*, Fig. 5. Then a second quantity of water is put on for another 24 hours, the water being again sent to tank *A*. Frequently, this water is slightly acidulated with hydrochloric acid, which gives better yields. The proportion of hydrochloric acid should not exceed 1 gallon to 250 gallons of water.

The cold, extracted meat is then sent to the cook room, where it is cooked for from 3 to 4 hours in the scalding tubs, or for 2 hours at 20-pounds-per-square-inch steam pressure in iron digesters. The residue of this cooking goes either into the tankage, or is occasionally sold for dog biscuit and other similar purposes; occasionally it has been worked into mince meat.

All the apparatus used in the manufacture of meat extract should be thoroughly cleaned every day. The tanks *A* and *B*, Fig. 5, should be kept scrupulously clean; not only the sides and bottoms, but most especially around the steam pipes. Occasional boiling with soda-ash solution cleans these vats perfectly. The coagulum obtained from these vats, in the sieve *d* and the filter bags finds proper use in the tankage. The filter bags must be thoroughly cleaned with hot water after each day's use. Occasionally, it happens that the liquid in the rotary evaporator turns cloudy, due to imperfect previous clarification. In such cases the liquid is conveyed into a steam-jacketed open kettle; either fresh cellar water or whipped blood is added to the liquid and the mass brought to a boil, producing a new coagulation, and by filtration through the bags a perfect clarification of the material is obtained.

55. Beef extract is put on the market in two forms, either as *fluid extract* or as *paste extract*, according to the degree of concentration.

Fluid extract is concentrated to a density of about 29° to 32° Baumé, and contains from 42 to 48 per cent. of moisture. Usually, there is an extra amount of common salt added, so as to bring the percentage of common salt up to 10 per cent., which requires usually from 2 to 4 per cent., there being from 6 to 8 per cent. of common salt in the extract, derived from the meat. The extra amount of salt in the fluid extract is necessary to give it better keeping qualities, no antiseptics ever being used in beef extracts. This extract is usually put up for the trade in glass-stoppered bottles; large quantities for storing, etc. are put up

in square, 14-pound tins, the cans being processed in dry steam for $1\frac{1}{2}$ hours at 10 pounds pressure per square inch.

Paste extract is boiled down to a concentration so that the product contains from 20 to 24 per cent. of moisture; it then contains from 55 to 65 per cent. of organic substances, and 15 to 20 per cent of salt. Paste extract is usually put up for the trade in porcelain or glass jars; but to store or ship in large quantities, it is put up in square, 14-pound cans and capped without any processing.

Fresh meat, such as roast beef, etc., prepared for canning purposes in the regular way yields about 1 pound of paste extract from 60 pounds of meat (from soup water and cellar water). By longer scalding in the preparation for canning, the yield can be raised to 1 pound of paste extract from 40 pounds of meat. If the meat is treated only for extract, that is, if the meat is finely cut, soaked for 24 hours with a slight addition of hydrochloric acid and cooked to thorough disintegration during 3 or 4 hours, the yield will be about 1 pound of paste extract from 25 to 30 pounds of meat. Bone liquors give $\frac{1}{4}$ to $\frac{1}{2}$ per cent. of paste from the amount of bones soaked in warm water. The labor in the beef-extract department (exclusive of the label room) is very small, one man and a helper being able to produce large quantities of extract.

MEAT CANNING

56. Introductory.—The meat-canning industry, in connection with the packing industry, was started in a small way in Chicago about the year 1877. It was primarily established with a view of saving a large proportion of the meat that had been up to that time either unsalable or of very little value.

The canning industry comprises so many different branches that in a paper of this character it is not possible to give all the details of all the operations. The most important features and the general plan and scope followed will be given in a more or less brief form.

The process used in the canning of meats is what is known as the **Appert process**, which was invented in 1809. Although this process has been well known to scientists for the entire period since its invention, its commercial application has, as before mentioned, been made available but a comparatively few years. Up to the present time, nothing has been discovered that has been able to supersede this universally applied process for the preservation of meats and of vegetables in cans.

The principle underlying the preservation of meat or other edible substances in air-tight cans is the fact that the decay of organic matters is not due alone to oxidation, but to the action of bacteria, organisms, ferments, etc., which attack and act on the organic substances, decomposing them and resolving them into their original elements or other compounds. The heating of the meat or other material in the process of canning destroys or paralyzes the bacteria and ferments, and sealing hermetically of the cans prevents bacteria from again entering into the material. Hence, canned goods made from good material and properly prepared remain indefinitely unchanged and in good condition. The process is, in fact, that of sterilization by heat.

57. Selection and Preparation of the Meat.—The meat selected for canning is ordinarily taken from lean cattle, which, while in good, healthy condition are not suitable for *beef* cattle. They are known in the trade as *canners*. These animals are slaughtered in a large measure to obtain meat for canning purposes. The parts of the beef that cannot be sold at a profit go to the canning department of the establishment. The portions sold in the fresh state comprise the hind quarters, loins, and, in some cases, salable fore quarters. The fore quarter furnishes the bulk of the meat for canning purposes. One reason for this is that there is less fat on this part of the animal.

The fore quarter is taken to the *boning room* and the bones removed entirely from the meat. The latter is cut into pieces of about 5 or 6 pounds each, in order to facilitate

curing uniformly throughout when placed in pickle for the making of corned beef. In boning, great care is exercised not to leave any bone in the meat, as even small bones are apt to break the stuffing machine and otherwise cause delay in stopping this machine when the meat is being stuffed into cans. The small pieces of meat are cured by themselves and when canning mixed with the larger.

58. Curing and Cooking the Meat.—The meat is cured in a mild pickle in which are placed small quantities of saltpeter and sugar, the former not exceeding 4 ounces to each 100 pounds of meat, and the latter varying in quantity from 1 to 2 per cent. The meat after remaining in this pickle from 15 to 26 days is cured and ready for boiling or shrinking.

After washing in cold water to remove the pickle, the meat is placed into boiling vats. These are wood-end vats holding, at each charge, about 700 pounds of meat. Different meats require different time of cooking. For canned roast beef, the meat is not subjected to the curing process, but is cooked, while fresh, in



FIG. 7

boiling water for 20 minutes, when it is removed and stuffed into cans by the can-stuffing machine shown in Fig. 7, after the requisite amount has been weighed off. The meat, by

cooking, suffers considerable shrinkage in weight. In *roast beef* this will average about 20 per cent.

For cooking *corned beef*, the meat is boiled for 10 minutes, after which it is allowed to stand in the hot water for 1 hour. This method freshens the meat and also causes far less shrinkage than by another common method of boiling it for 1 hour. The average shrinkage in cooking corned beef is about 38 per cent., although at times it will not be more than 30 per cent. The water (soup water) from the cooking or shrinking of meats is used both for making soups and also in the manufacture of beef extract.

59. Canning and Processing. — When the meat is removed from the cooking tubs, it goes to the cutting table,

FIG. 8

where it is cut suitable for the stuffing machine; it is then passed to the weighing table, where the proper amounts are weighed off and fed to the stuffing machine. After the cans are stuffed, they are reweighed, wiped, the cap placed over the opening through which the meat was stuffed, and inspected. The cap is soldered on when the can is on the capping machine shown in Fig. 8. The small hole in the center of the cap, the vent, is finally closed with solder, when the can is ready for processing. This may be

done either in a water bath or in a retort like that shown in Fig. 9, the latter, or steam, process being most commonly used.

The retort shown in Fig. 9 is 28 inches wide, 32 inches high, and 10½ feet long, and will hold at one time 700 3-pound cans, when arranged on the iron trays as shown in the illustration. The retort is provided with steam connections and a steam gauge for registering the steam pressure. Doors

FIG 9

are at each end, provided with the usual tightening appliances. There is always a valve controlling the exhaust of the retort, as with some goods and in "boiling off" sealed cans the exhaust is kept open. The retort has a perforated steam pipe fastened to the entire length of the bottom, to equally distribute the steam throughout the box when processing.

Different materials are subjected to different pressures of steam and remain in the retort for different periods of

time. The size of the cans also influences the time and pressure. As a general average, the cans are subjected to a temperature of about 220° F. for 2 hours.

The object of processing, as before mentioned, is for complete and thorough sterilization, as well as for the additional cooking of the meat. At the expiration of the required time in the retort, the cans are removed on the trays and punctured (the vents opened) in order to allow the air in them to escape. This operation is done as quickly as possible, the vents closed up, and the cans again returned to the retort and heated again for a shorter or longer time, depending on the size of the can. This latter processing is technically termed *boiling off*.

Processing and boiling off under water with closed vents is also practiced extensively with satisfactory results, and it is claimed by some that this method gives a better flavor to the canned product. In processing in the water bath, the time of process is taken from the time the water boils. These process tanks, either of wood or iron, are 3 feet 10 inches long, 2 feet 10 inches wide, and 3 feet 6 inches deep. The cans are put into the process tanks on trays, to facilitate handling in removing. The cans when removed are also punctured the same as in steam processing, sealed, and boiled off again in the water.

The cans of meat after removal from the retort or process tanks the last time are taken to a room, the *shower room*, where cold water is run on them until they are cool. This sudden chilling contracts the ends of the cans, making them slightly concave, which is the proper condition for canned goods. They are then sent through a washing trough by mechanical means, to remove any grease or other accumulations from the cans. From the washing machine the cans go to the lacquering and labeling room, where they are finished and made ready for packing and shipment.

60. Specifications for Canned Beef for the United States Navy.—The specifications for canned beef for the United States navy show what may be regarded as the

requirements in general for canned beef, the regulation style of cans, methods of packing, etc. They are as follows:

“Shall be of the best quality ox beef, full weight, in tins of 2 pounds each, the weight of tins not included. The side of the tins to be made of best double (XX) I. C. tin and the ends to be single (X) tin. Each tin to be marked with contents, name of contractor, name of packer, brand, and date of packing, and to be properly lacquered. Each dozen tins of beef to be enclosed in a substantial box, made of well-seasoned pine, properly nailed, the sides to be $\frac{3}{4}$ inch and the ends 1 inch thick. The top of each box to be securely fastened with nails of suitable size and length, and to be marked with the contents, name of contractor, and date of packing. The contractor shall guarantee that the beef shall keep good in any climate for 1 year from the date of delivery.”

BUTTERINE

61. General Remarks.—Butterine, or oleomargarine, is composed of oleo oil, neutral lard, cottonseed oil, and genuine butter, with a small proportion of moisture, salt, and coloring matter.

The manufacture of this product is carried on in packing houses where State statutes permit its manufacture. This article has to a large extent replaced natural butter with people of limited means. Contrary to the general impression, butterine is made from the highest grade material and, aside from the natural butyric and other flavors, is almost identical in composition with genuine butter. The highest grades of butterine have as much as 33 per cent. of genuine butter in their composition and others 25 per cent. This proportion may be and is varied at will by the different manufacturers. Each manufacturer has his individual formula, but as every factory in this country is under direct government supervision, it is obvious that only materials of unquestioned healthfulness may be used.

Each factory is required to make a daily report of the exact quantity and kind of materials used and also the number of pounds produced from this material. This report must be made under oath to government officials, and severe penalties are incurred for infractions of the regulations.

The manufacture is attended with the most scrupulous cleanliness, not only as a matter of personal convenience, but as an essential in producing a merchantable article of trade, as without this cleanliness good butterine cannot be made.

62. Ingredients Used in Butterine.—As previously stated, the quality of the ingredients used in making butterine is of the highest. Their condition and preliminary treatment is as follows:

The **milk** used is received in cans surrounded by felt casings, to keep it from changes of temperature. It is poured into a receiving vat, from which it is pumped through a fine strainer, to remove all impurities, to the centrifugal separator. In this machine the cream is separated from the milk at a temperature of 80° F., the milk being of no further service for the butterine maker. The cream flows through block-tin troughs to a Pasteurizer, where it remains until Pasteurized. The temperature used here is 170° F.

The **cream** after this process is allowed to ripen, i. e., acquire acidity, for 24 hours at a temperature of 60° to 70° F., when it is ready for mixing with the other ingredients for churning.

The **neutral lard** used for butterine does not require to be grained, as it is used in a molten condition. The same applies with equal force to the oleo oil.

The **cottonseed oil** used is of the grade known as *butter oil*—the choicest grade of this material.

63. Churning and Finishing the Butterine.—The churns used for producing butterine are fixed, upright, sheet-iron cylinders capable of holding a charge of about 600 pounds of mixed material. The churns are partly enclosed on the

top with curved sheet iron. Inside the churn are the paddles fixed to a revolving shaft, and when churning make from 500 to 600 revolutions per minute.

The proportions of the different ingredients, as previously mentioned, vary. The following proportions for 100 pounds of butterine are those used by a manufacturer making about 15 tons of butterine daily:

Cream sufficient to produce 15 pounds of butter; cottonseed oil, 15 pounds; neutral lard, 35 pounds; and oleo oil, 35 pounds. The proportion of butter color varies with the season and trade. From $\frac{1}{2}$ to 1 ounce per 100 pounds of butterine is the usual amount added. This is placed in the churn with the other ingredients at the time of churning.

The desired weights of the various ingredients are run into the churn and the mixture churned by means of the revolving paddles for 6 or 7 minutes, when the operation is completed. The best temperature for churning is about 90° F., and the various ingredients are introduced into the churn at temperatures to obtain this. The oleo oil and neutral lard are run in at about 110° F., the cream at about 70° F., and the cottonseed oil at normal room temperature.

The butterine is now run from the bottom of the churn into tanks or vats of ice-cold water, to produce the requisite butter grain. A constant supply of ice is in the water to keep it cold. Some manufacturers place cakes of ice in the bottom of the vats, over which the water flows constantly, thus maintaining a steady temperature of about 34° F. The churned product is allowed to remain in these vats with the water for about 10 minutes, or until it has acquired a firm *set*, when it is thrown by means of wooden shovels on inclined tables to drain. The temperature of this room is kept at 70° F. At this stage the butterine is salted with an amount varying with the requirements of the trade. From $\frac{1}{2}$ to $\frac{3}{4}$ ounce to each pound of butterine is sprinkled over the mass, which during the 10-hour period of draining is turned over three or four times to allow the water to drain from it.

This is generally practiced at night, so that the material is ready for working the next day, when the butterine is

worked with the regulation butter worker, to press out the superfluous water and to evenly distribute the salt. A very small amount of sugar, from $\frac{3}{4}$ to 1 per cent., is usually mixed with the salt for the purpose of "toning down" the harsh taste of the latter.

64. A peculiar fact in connection with the butterine industry is that the manufacturers almost universally employ Ashton salt, an English product, to salt their butterine. It is claimed that a far better product can be made by the use of this salt than by the use of any domestic salt. Butterine is packed in regulation butter tubs, upon every package of which is placed the usual revenue stamp and a penalty label. It is also made into pound prints resembling butter and wrapped similarly in parchment paper. The labor attached to the making of butterine is very small, a few employes being able to produce large quantities daily.

GLUE

65. General Remarks.—Glues are now made in many packing houses from raw material that was at one time sold to glue manufacturers and from liquors formerly wasted. While perhaps not properly belonging to the packing industry, a short account of the method of making glue will be given.

The chief sources of glue in the packing house are the waters or liquors from the boiling of cattle and sheep heads, feet, and bones, from sinews, hide trimmings, horn piths, calves' heads, and pigs' feet. In making glue, a thorough knowledge of the raw material is necessary in order to produce good results. In this manufacture, as in all others, poor material will never yield a high-grade finished product. Material in prime condition, if allowed to lie around in heaps or be subjected to incipient decomposition, will very quickly become unfit for use as glue; or if used, will produce

only low-grade, weak, and foul-smelling glues. To obtain good glues, it is necessary to have the stock in a sound, sweet condition.

66. Glue Stock.—Glue stock may be either “green” or “dry,” “green-salted” or “dry-salted.” Green stock consists of material in the fresh state, such as fresh hide pieces, sinews, calves’ heads, feet, etc. Dry stock consists of glue-making material, such as bones, etc., dried, without salting or other treatment. Green-salted material comprises fresh stock that has been cured by means of salt, such as pieces of salted hides, sinews, calves’ trimmings, etc. Dry-salted goods consist of trimmings, etc. from South American hides, rawhides, etc.

In examining glue stock for utility, the examination should include, for dry and dry-salted stock, the gain in weight by soaking over night in water. This should be about 50 per cent. of their original weight, and in addition the soaked pieces should be tough and of firm texture. The odor of both the soaked stock and the water should not be strong. If such is the case, the stock is moldy or the gelatinous matter has been destroyed by insects. The impurities, consisting of dirt and salt, should not exceed 5 to 6 per cent.

Green-salted stock should be examined for the purpose of determining if it contains any decomposed, discolored, slimy, or heated pieces. The latter if not in a state of decomposition are bordering upon it. The total amount of salt and moisture for this material should not exceed 40 per cent.

The bones in the packing house designed for glue should not be overcooked; that is, they should be smooth and hard and not have a chalky, white surface. The latter condition indicates that most of the glue has already been cooked out of them. Soft bones, such as rib bones, yield more glue than the hard bones of the legs and thighs. Bones that have been exposed to weather influences were formerly supposed to yield but little glue of poor quality, but by a

recently devised secret process, these bones may be made to yield an excellent light-colored glue.

Horn piths should be free from the skin covering and should not be discolored from drying on steam coils. If the tips of the piths are easily broken off, it shows that they have been subjected to a very high temperature, and much of the gelatinous matter thereby destroyed.

Fresh glue stock presents no difficulties in the manufacture of glue. But care must be taken to keep it from becoming decomposed before using.

67. Bone-Glue Liquor.—One of the chief products of the packing-house glue department is bone glue, of which there are many qualities, varying from a fine white gelatine to dark-colored, low-grade glues. One method of preparing this product consists in cooking the bones in open vats and evaporating the resultant glue liquors to the desired density in the vacuum pan, from which the liquors are run into the forms for cooling. As many cookings of bones for glue liquor are made as is practicable; that is, as long as the liquors obtained contain sufficient glue to make evaporation profitable for forming the glue jelly.

Another method of producing glue liquors is by cooking the bones under pressure, as described later. The same principle is applied in other cases; such, for example, as cooking the bones in tanks under 10 or 15 pounds pressure per square inch for two periods of 2 hours each. The yield of glue by this method is better than that obtained from cooking the bones in open vats. From the pressure method, from 11 to 14 per cent. of glue is obtained, while the open cooking yields but from 6 to 10 per cent. When cooking the bones in the tanks under pressure, water sufficient to cover them must be added.

Bones are sometimes leached with acid, to dissolve from them the inorganic matter. For this purpose the bones should be as free as possible from grease, and also tough and hard. The bones are covered with dilute hydrochloric acid. They remain covered for a length of time, depending

on the size of the bones, generally from 3 to 4 weeks. The bones after this leaching are soft and spongy and have a rather strong odor. The material is then known as *raw gelatine*, and is suitable for the best grades of bone glue and gelatines.

The glue liquors while hot are run to the settling tanks, where they are allowed to settle and clarify. Any grease appearing on the surface is carefully removed, as it is important to have all glues as free from grease as possible.

68. Horn-Pith Glues and Gelatines.—In using horn piths for making glues and gelatines, they are leached with hydrochloric acid of 2° Baumé or with phosphoric acid of 6° Baumé in vats with perforated false bottoms. When the horn pith can be cut through easily in any direction with a knife, the leaching is finished and the horn piths are then drained free of acid, which is replaced with clean, soft water until all traces of acid are removed. This material furnishes fine gelatines and glues upon cooking. From 30 to 35 per cent. of raw gelatine may be obtained from dry horn piths; and from the dry, raw gelatine about 90 per cent. of glue. When phosphoric acid is used for leaching, from 6 to 8 weeks are required for leaching the horn piths.

69. Head-Glue Liquor.—The glue liquors from cooking heads and feet, previously described, yield upon evaporation light, yellowish-colored glues, closely approaching white. The liquor upon drawing off from the cooking vats has a density of about 2° Baumé. It is allowed to clarify by settling and is then evaporated in the vacuum or other apparatus to the desired consistency, when it is run into the jelly molds, cooled, cut, and dried in the usual way.

70. Clarification of Glues.—When glue liquors are made from sound stock, they come from the cooking vats in a clear condition and, upon standing a few hours in the settlers, become easily clarified, any impurities rising to the top with the grease or settling to the bottom. The glue

liquor after settling is tested for clearness and, when in a satisfactory condition, is evaporated either in the vacuum pan or other apparatus and run into forms for glue jelly.

The first runs of glue liquors in general are easy to manipulate, but the last runs, containing the residual glue stock, are usually muddy and contaminated with impurities. This class of material is most frequently made into colored glues by mixing zinc oxide with them, thus producing a good-looking, merchantable article.

It is necessary for glues to have an acid reaction, in order to clarify by settling. Where a neutral or alkaline condition exists, it is better to clarify by means of a solution of alum sprinkled into the glue solution. Albumin is used at times as a clarifying agent with good results. The glue liquor is cooled to about 140° F., when the solution of albumin is added and the whole stirred thoroughly and then heated just short of the boiling point. The albumin, coagulating, entangles the impurities in the glue liquor and settles to the bottom. The addition of a small amount of ammonium chloride with the albumin makes the clarification more complete.

In the clarification of glues, the use of the filter press has of late years been brought into requisition. If a decolorizing material, such as bone black, be mixed with these glue liquors, upon pumping the mixture through the filter press, a clear, brilliant, and somewhat bleached product is obtained. This method has superseded in many establishments the old way of allowing the glue to clarify itself by settling, on account of the time saved and the reduced liability in warm weather of the warm glue liquors becoming *sour*, or decomposed, and thereby useless.

71. Bleaching Glue Liquors.—Bleaching of glue liquors is largely practiced by modern glue makers. Bones, before cooking, if covered with a 1-per-cent. solution of sulphurous acid for 24 hours, are bleached to a considerable extent. Such bones are washed free of acid and when boiled furnish a very light-colored glue.

A very common method of bleaching glue liquors is to pass washed sulphur-dioxide gas through them until the desired light color is obtained. This is the most common and the cheapest method for bleaching. Liquid sulphur dioxide is now manufactured for this purpose, and is transported in heavy iron cylinders similar to those used for anhydrous ammonia. While the sulphur-dioxide method of bleaching is cheap, it cannot always be used with good results on very dark-colored glue liquor. Other bleaching agents employed are zinc salts, such as sulphate and chloride, but, unless carefully used, their presence in the glue is liable to be very detrimental for certain purposes.

In Germany, limed glue stock is treated with hydrochloric acid and chloride of lime for about $\frac{1}{2}$ hour, after which time it is thoroughly washed. If this treatment is continued too long, however, the stock will become hard and almost insoluble. The so-called Cologne glues, which are very light-colored, are said to be produced by this treatment. Peroxide of hydrogen has been used for bleaching glues and answers the purpose well. It will turn the darkest brown glue liquors into a light-yellow color almost instantly. The item of cost, however, has prevented this material from assuming a commercial importance.

72. Preservatives Used in Glues.—Preservatives are frequently used in glues. Sulphur dioxide, in addition to bleaching glues, also acts as a preservative for them. The preservatives most commonly employed are the zinc salts—the oxide, sulphate, or chloride. At the same time, the former has the additional advantage of producing a light-colored glue. Alum is also employed as a preservative, but it is liable to give a flaky appearance to the glues if used in too large quantities. Formaldehyde has of recent date been employed as a glue preservative. Its use for this purpose is too recent to allow of any conclusions as to its effectiveness or feasibility. If, however, an excess over and above that necessary for preservative purposes is added to glue liquor, the glue will be rendered insoluble almost immediately.

73. Concentration of Glue Liquors. — Glue liquors may be concentrated by evaporating them in jacketed kettles, in evaporating vats provided with steam pipes, or by means of a revolving steam-heated drum, similar to Fig. 6. With all these methods, the evaporation takes place under ordinary atmospheric conditions. The results in many cases are very satisfactory, but all open evaporators have the disadvantage of having the glue liquors subjected to a high temperature, which is greatly detrimental to the strength of the finished glue. With many of these evaporators, the heated iron surface reaches a temperature of nearly 300° F., which naturally scorches the glue, forms a crust on the heated surface, and thereby hinders a rapid evaporation. The glues produced by open evaporators do not compare favorably in strength and color with those made from the same kind of material by evaporation in a vacuum apparatus.

The concentration of liquors in the vacuum apparatus is accomplished by producing a partial vacuum in the apparatus, thereby partially removing the atmospheric pressure from the liquid placed therein. By exhausting the air and producing a vacuum of 8.7 pounds per square inch, as shown by the vacuum gauge, the liquor enclosed in the vacuum pan will boil at a temperature of 170.1° F. The lower the temperature at which any glue is made, the better will be its color and strength. This is especially true of high-grade glues.

Vacuum evaporators are operated either by direct steam or by exhaust steam of an engine. Another advantage in the use of this apparatus is that less fuel is required for the evaporation of a given weight of water. In the ordinary vacuum pan, 1 pound of coal will evaporate upwards of 8 pounds of water, while with open evaporators an excellent use is made of the coal if 1 pound evaporates 6 pounds of water. With a vacuum pan of three "effects," or chambers, an evaporation of 24 pounds of water is obtained from 1 pound of coal. The economical principle of the multiple-effect vacuum pan consists in utilizing the heat over and

over again. The steam introduced into the tubes of the first effect evaporates a quantity of water and the steam thus formed passes into the tubes of the second effect. The steam produced by this evaporation passes into the third effect and evaporates still more water. It has been demonstrated, however, that more than four effects cannot be

FIG. 10

used with satisfactory results. Fig. 10 shows a modern multiple-effect vacuum apparatus of three effects.

The single effect vacuum pan is used where there are comparatively small quantities of liquor to be evaporated. But in the largest packing houses, the multiple-effect vacuum pans are employed in concentrating glue liquors.

74. With either kind of apparatus, open or vacuum, the clarified glue liquors are evaporated to a thick consistency,

so that they will set to a firm jelly within 10 or 12 hours. The concentrated glue liquors are run from the evaporating apparatus into molds, or boxes, for forming the desired cake of jelly. These molds are usually made of galvanized iron and hold about 50 pounds of the glue liquor. They are made flaring, so that the glue jelly may be easily emptied from them. Any desired shape may be used, as there is no general standard as to their shape.

The boxes are filled to within $\frac{1}{2}$ inch of the top and are allowed to set in a well-ventilated room protected from extremes of temperature until the jelly has become hard and firm. Some establishments set the boxes in a trough of cold, running water and allow them to be cooled in this way. Under no circumstances should the glue liquor be allowed to freeze or to remain too long in a moderately warm temperature. In the former case, the glue jelly is brittle and cannot be cut, and in the latter, the liquor will decompose and become worthless. The jelly in the boxes, when firm and in condition to be handled, is ready for cutting into sheets.

A patented process is in use in one large factory, not a packing house, for cooling, setting, and cutting the glue by a continuous operation. The glue liquor is chilled on a revolving, brine-cooled cylinder, which, revolving slowly, dips the lower part into the glue liquor and carries sufficient of it on its surface to form, within one revolution, a thin layer of firm jelly. This can be wound off the cylinder as an endless ribbon, cut by the machine into suitable sizes, spread on frames, and sent to the drying room.

75. Cutting Glues.—The glue jelly is usually cut into sheets by wire machines, although other kinds are used. The wire machines consist of frames of steel with wires so set as to give a slice of glue jelly of the desired thickness. In the old-style machine the jelly is cut by movable wires; in the new style the wires are stationary, being set in steel frames. These frames are about 2 feet apart from one another, in line, so that when the cake of jelly moves on an

endless belt, each wire in turn cuts a slice of jelly. For cutting low-grade glues a knife-cutting machine is used. The circular knives are set on a shaft sufficiently far apart to make the desired thickness of the sheet of jelly. With this machine, the tops with the bubbles and the bottoms with the settlings are removed and remelted with fresh material.

The sheets of glue after cutting are spread on nets of galvanized-iron wire, with meshes of about 1 inch. These nets have a framework of wood which sets into another frame built on a truck. As fast as the nets are covered with sheets of glue they are placed on the truck. The latter when filled is run into the drying room and there the glue remains until dry. Although perfectly dry to the touch, it ordinarily contains from 10 to 13 per cent. of moisture. Excessive drying of the glue is not desired by the manufacturers for very obvious reasons. In a heated chamber furnished with fans or blowers, the drying operation occupies at the present day but a few hours, where formerly it consumed days, by exposure to drying in airy lofts.

Sheets of glue that are imperfect or broken may be made, if of cheap quality, into ground glues by passing the material through a grinding mill. Where the glue is of high quality, imperfect sheets and pieces are remelted with the next batch of high-grade material. The lighter the glue, other things, such as strength, clearness, etc. being equal, the better the price obtained for it.

76. Yields of Glue.—From green-salted hide trimmings, sinews, etc., from 18 to 20 per cent. of glue may be obtained. These materials, dry, yield from 50 to 65 per cent., according to quality and condition. From hard, dry bones, an average yield of 18 per cent. is obtained. From green rib bones—practically the only way this material is worked up for glue—an average yield of 12 per cent. of glue is obtained.

PACKING-HOUSE INDUSTRIES

(PART 8)

VARIOUS ANIMAL PRODUCTS AND THEIR DISPOSITION—(Continued)

CURED MEATS

1. Introductory.—The curing of the meat products of the packing house is such a wide subject that it cannot be treated in full in the present treatise. The principle underlying the curing process, either in the wet (pickle) or dry (salt) way, is the prevention of putrefaction of the products by means of salt and other curative agents. Among the latter are saltpeter, borax, and boracic acid. Salt alone may be used, but to obtain the best results for mild cures, its harsh effect is toned down with sugar, sirup, or molasses. When these saccharine substances are used in conjunction with brine or dry-salt curing, the meats are termed sweet-pickled meats.

2. Dry-Salt Meats.—These are meats that have been cured by covering with salt, and piled about 6 feet high on the floor of the curing cellar. A small proportion of saltpeter is frequently mixed with the salt, to give to the lean meat the ruddy appearance always desired. These cuts of meat are taken down and repiled three or four times during the period of curing. This operation is technically

termed *overhauling*, the object being to allow the salt and brine, made by the moisture in the meats, to affect all parts of the cut equally. The time allowed for curing these articles in packing houses is based on 1 day per pound of the weight of the cut. The dry-salt method of curing is most largely and almost exclusively applied to pork products. Dry-salt meats are shipped for domestic trade in bulk; and for export trade, packed in borax in boxes.

3. Wet-Cured Meats.—Both pork and beef products are cured in *brine* or *pickle*. The distinction between these terms is that the former is a solution of salt in water, while the latter is brine, having added to it various ingredients, such as sugar, borax, boracic acid, Glauber's salts, and saltpeter. In rare cases saccharine has been substituted for sugar or sirup, but its use at the present time is not extensive, owing to its unknown ultimate effect upon the meats.

Each establishment has its own formula for pickle and its own method of curing in it. The time required for curing meats in pickle varies according to many circumstances, such as the strength of the pickle (percentage of salt) and the size and nature of the cut. While a piece of bacon weighing 4 or 5 pounds may be cured in 21 days, a very large-sized ham may require over 100 days, all the pieces of meat being subject to frequent overhauling.

Small pieces of beef to be used for canning purposes are cured in 15 days, as by their small size—4 to 6 pounds—they are readily permeated by the pickle. Even then, as with all pickled meats, they require frequent disturbance or transfer, to insure a uniformly cured product.

4. Smoked Meats.—After coming from the pickling process, hams, bacon, and some other cuts are subjected to smoking as a further means of assisting in the preservation of the products. The object of putting the meats into smoke is twofold—first, to evaporate a portion of the moisture of the meat and, in the next place, to impregnate the

products with creosote and other empyreumatic compounds, the latter giving to smoked meats their characteristic flavor.

The meats are hung in a smoke house—an enclosed compartment, at the bottom of which slow, smoldering fires are kept burning. Hard woods are used for fuel to produce the required smoke, as soft woods contain too much resinous matter, which produces ruinous effects on the meat. A small quantity of sulphur is sometimes burned to bleach the smoked meats.

The products remain in contact with the smoke for a period depending on many conditions, which are judged from personal observation. Among these are the depth of color desired, size of pieces, temperature, etc. A shrinkage of weight always takes place in this process, which depends also on many circumstances; a general average of loss in weight may be taken as from 12 to 14 per cent.

The smoked products are removed from the smoke house and allowed to cool at the natural temperature of the room, when they are ready to be sold or to be packed for shipment.

BLOOD ALBUMIN

5. Preparation.—Blood albumin is one of the products made from the blood of slaughtered animals. The blood as it comes from the animal is caught in shallow pans, each just large enough to hold the amount from one animal. It is then allowed to rest without stirring or agitation until it clots. It is an essential part of the process of manufacture that the blood should not be stirred or it will be defibrinated and the proper separation cannot be made.

After clotting, the clot is scored by cutting up with a sharp knife into small squares and placed in shallow pans with perforated bottoms, through which the serum or albumin drains from the clot. The dark-red clot is used for the manufacture of the ordinary dried blood. When separated

from further impurities and dried under suitable conditions, the serum that drains through is the product that forms the dried albumin of commerce. The serum at this stage of the process is of a pinkish or light-red color, due to the presence of some red corpuscles that have been imperfectly separated by clotting.

6. The next operation consists of the separation of these red corpuscles, as the quality of the finished product depends on the perfection of this separation. The serum is placed in shallow pans, which are about 3 feet long by 18 inches wide and 4 or 5 inches deep. At the center of the bottom of these pans is a perforated rubber stopper, through which a $\frac{1}{4}$ -inch glass or lead tube extends until it reaches just above the surface of the liquid. The pans are filled with the serum and allowed to stand in a cool place for a few hours, during which time the red corpuscles begin to sink. As the liquid clears at the top and the corpuscles sink lower and lower, the tube is gradually lowered to draw off the clear serum into suitable vessels placed underneath, this operation being performed at intervals as long as the liquid runs off clear. When it is seen that further continued settling will no longer produce a clear liquid, the receptacle is changed, and that which afterwards comes over is used in making inferior grades of the product.

The serum that has drained off clear is of a very delicate pink color. It is placed in small, shallow, smooth enameled plates, resembling ordinary pie plates, each of which holds about $\frac{1}{2}$ pint. Previous to filling, the plates are slightly greased. They are then placed in a drying room and kept at a temperature of about 110° F. until the product dries. The product is then scraped off the plates, which operation is easily accomplished, as the film of grease prevents the dried albumin from adhering to the plates.

It is important that the temperature of the drying room be kept as low as possible and yet dry the product in a few hours. Otherwise, there is danger of the serum coagulating and thus ruining the product. The darker colored albumin

is dried in the same manner as the foregoing. The first and best grade is a very clear and pale amber-colored product, its quality and value depending very largely on its lightness in color. Blood albumin finds an extensive use in the textile industries in printing cotton fabrics.

7. As liquid blood is a very perishable product, it is necessary that all the operations up to the drying should be performed at a low temperature, to avoid decomposition and consequent inability to make albumin from the blood. It was customary in former times to manufacture the product only in winter. With the modern system of artificial refrigeration, however, it is now possible to turn out a uniform product at all times, independent of the seasons.

In the very latest and most approved methods of manufacture, the separation of the serum is accomplished by centrifugal machines and the evaporation subsequently conducted at a low temperature in a vacuum apparatus. Most of the product on the market, however, is still made by the old process, but it will undoubtedly soon be superseded by the new methods.

The yield of blood albumin from blood is, theoretically, 50 per cent., but it is never realized practically. Five bullocks yield about $4\frac{1}{2}$ gallons of serum, which when dried give $4\frac{1}{2}$ pounds of albumin. The blood of 20 sheep or 34 calves gives the same quantity of blood albumin.

DIGESTIVE FERMENTS

8. **Introductory.**—The known digestive ferments all belong to the class of soluble, or unorganized, ferments. They are sharply distinguished from the insoluble, or organized, ferments, of which the type is yeast, in not having the power of self-nutrition and self-multiplication. In composition, digestive ferments resemble proteid substances (see *Organic Chemistry*), and contain carbon, hydrogen, oxygen, and nitrogen in somewhat similar proportions to albumin. None of these ferments has yet been obtained in a state of

absolute purity. All digestive ferments are soluble in water and all are diffusible, though with difficulty, through animal membranes and parchment paper. They are precipitated from their watery solutions by absolute alcohol, but, unlike other proteids, with the exception of peptone, they are not truly coagulated by alcohol. When the alcohol is removed, the ferments are still found to be soluble in water and to retain their activity unimpaired. All digestive ferments are coagulated and rendered permanently inert by the heat of boiling water; and when in solution, they are coagulated and destroyed by a heat of about 160° F.

The digestive ferments produced in the packing house are mainly pepsin and its products, the former, however, being most commonly made. Beef peptone is made by but two large packing houses in this country.

9. Pepsin.—Pepsin is prepared from the mucous membrane of the stomachs of hogs, although it may be prepared from the stomachs of other animals. The large and steady supply of material from hogs makes it most conveniently available. The stomachs are emptied of their contents and thoroughly washed in cold water. What food clings to the mucous membrane is removed by hand labor. In the washing, violent, energetic motions should be avoided, or a great deal of the pepsin-containing membrane is liable to be removed mechanically. Gentle handling is essential in the first stages of the preparation. The outsides of the stomachs are trimmed away.

That portion of the stomach reserved for the pepsin is chopped into small pieces and placed in water acidulated with from 3 to 4 per cent. of pure hydrochloric acid. The receptacle used for this purpose in the packing house is usually a large open-head hogshead. The material is allowed to remain in this weak acid solution, kept at a temperature of 104° to 122° F. until it undergoes self-digestion. This operation is materially assisted by frequent stirring, but from 36 to 48 hours, and sometimes longer, are required for the solution to be effected. At this stage of preparation,

the liquid is very prone to decomposition, especially in the presence of ozone, occasioned by thunder storms, and great watchfulness is required. An innocuous antiseptic condition is occasioned by passing sulphur dioxide into the solution from a generator, until the solution smells strongly of the gas. This operation serves at the same time to bleach the product.

In this condition, the liquid is allowed to stand and clarify itself by the precipitation of the mucous, without causing any material injury to the pepsin from putrefactive changes. The resulting clear liquid is decanted or drawn off, and to it is then added common salt, the temperature of the liquid being maintained at 94° F. until complete separation of the pepsin results by precipitation. The pepsin thus obtained—the floating scum—when collected, pressed, and dried constitutes *crude pepsin*, which even in this form is marketable, being very active and meeting certain requirements of trade. The product has a faint but not disagreeable odor, a brownish-yellow color, and a slightly saline taste.

10. Purified Pepsin.—The above product, preferably but not necessarily in the moist state, is dissolved again in weak hydrochloric acid, and the solution thus obtained subjected to dialysis by any suitable means until the salt has been eliminated from the pepsin solution. The purified liquor is then concentrated, preferably in vacuum apparatus, care being exercised not to have the heat exceed 100° or 105° F. The concentrated solution is then dried on glass plates, the edges of which are raised to hold it. These plates are about 15 inches wide and 20 inches long, with the projecting edge $\frac{1}{4}$ inch high. The thin layer on the plates is dried as rapidly as possible in a drying room arranged with shelves to hold a number of plates. The temperature should not exceed 102° F. in this room, which must be well protected from dust. When thoroughly dry, the pepsin (now the so-called pure pepsin of trade, known as *scale pepsin*) is scraped from the glass plates. It will have a digestive power of about 1 to 3,000; that is, 1 part of pepsin

will digest 3,000 parts of freshly coagulated egg albumin, if the operation has been properly performed. By further redissolving and dialyzing, the digestive power may be greatly increased, but ordinarily the price obtained is not commensurate with the labor and expense involved. The above strength—1 to 3,000—is the standard requirement for pharmaceutical and medical purposes.

11. Powdered Pepsin.—The scale pepsin made by the above process is ground in any suitable mill in which the material is protected from the atmosphere during the process of grinding. This is necessary, as the scale pepsin is so hygroscopic in its nature that attempts to powder it without this precaution will prove futile. Several suitable mills for this purpose, readily available and very serviceable, are on the market. Powdered pepsin is used for making into pills, tablets, etc., and has the same digestive power as the scale pepsin from which it is made.

The compound preparations of pepsin enter into the field of the pharmacist rather than that of the chemist, and their preparation will not be treated here.

12. Yield.—The yield of pepsin varies with the class of hog stomachs used. From a test of 3,318 pounds of membranes, trimmed for pepsin making, a yield of 117 pounds of high-grade pepsin was obtained. The weight of membranes from 250 whole hog stomachs was 200 pounds, thus giving a yield of 2.8 pounds of pepsin for each 100 stomachs. Another test from 9,000 stomachs gave a yield of 162 pounds, or 1.8 pounds of pepsin for each 100 stomachs.

The labor attached to the manufacture of pepsin is very slight. Ordinarily, the pepsin maker, with a boy, accomplishes the entire manufacture at a comparatively small cost.

Pepsin in dry form, in common with all digestive ferments, permanently retains its properties. What is true of pepsin is true of all digestive ferments in characteristics of this nature. Moisture and heat are favorable to their decomposition and, as an essential quality, they should be so prepared

as to have peptone and all other substances of hygroscopic properties eliminated as completely as possible. The digestive ferments remain inert, but are not injured in the least by a low temperature. Pepsin exerts its activity in acid solutions only, while pancreatin acts best in neutral or alkaline solutions.

13. Valuation of Pepsin.—The chemist is frequently called on to determine whether a pepsin meets the requirements of the standard strength. The most common and approved method of testing is that described as the official method in the United States Pharmacopœia of 1890. It is carried out as directed below. For this test the following solutions are necessary:

(a) To 294 cubic centimeters of distilled water add 6 cubic centimeters of dilute hydrochloric acid, the latter to be made by adding 2 parts of water to 1 part of strong acid.

(b) Dissolve .067 gram of the pepsin to be tested in 100 cubic centimeters of solution (a).

(c) Add 5 cubic centimeters of solution (b) to 95 cubic centimeters of solution (a) brought to a temperature of 40° C. The resulting 100 cubic centimeters of liquid will contain 2 cubic centimeters of dilute hydrochloric acid, .00335 gram of the pepsin to be tested, and 98 cubic centimeters of water.

The test is made in the following manner:

Immerse and keep a fresh hen's egg in boiling water for 15 minutes; then remove it and place it in cold water. When it is cold separate the white, coagulated albumin and rub it through a clean sieve having 30 meshes to the linear inch. Reject the first portion passing through the sieve. Weigh off 10 grams of the second and cleaner portion, place it in a flask of the capacity of about 200 cubic centimeters, then add one-half of solution (c) and shake well so as to distribute the coherent albumin evenly throughout the liquid. Then add the second half of solution (c) and shake again, guarding against loss. Place the flask in a

water bath, or thermostat, kept at a temperature of 38° to 40° C. (100.4° to 104° F.), for 6 hours, and shake it gently every 15 minutes. At the expiration of this time the albumin should have disappeared, leaving at most only a few thin, insoluble flakes. Trustworthy results, particularly in comparative trials, will be obtained only if the temperature be strictly maintained between the prescribed limits and if the contents of the flasks be agitated uniformly and at equal intervals of time.

The relative digestive power of pepsin that is stronger or weaker than the above, may be determined by ascertaining, through repeated trials, how much of solution (*b*), made up to 100 cubic centimeters with solution (*a*), will be required to exactly dissolve 10 grams of coagulated and disintegrated albumin under the given conditions. The official strength of pepsin for pharmaceutical uses, where it is most largely employed, is that it must be capable of digesting not less than 3,000 times its own weight of freshly coagulated and disintegrated egg albumin when tested by the process given above.

14. The necessary apparatus for manufacturing pepsin and compound preparations of it includes the following:

Mill, sifter or bolter, percolators, funnels, glass plates for drying, glazed stone jars, the necessary hogsheads, barrels, etc.; all of which may be purchased for an amount not exceeding \$400.

For making the pepsin tablets, pepsin glycerol, essence, wine and aromatic pepsin, a hot-water bath, hot-water funnel, and several glass bottles and funnels are necessary. This additional apparatus, sufficient for a large daily output, may be obtained for \$350 additional, making the total for a complete pepsin manufactory about \$750.

15. Peptone.—By allowing the solution from which the pepsin precipitate has been removed to cool gradually, the salt will crystallize out. This solution contains practically all of the peptones (see *Organic Chemistry*), and after the

salt has crystallized from it the peptones are left in solution, from which they may be obtained by evaporation and drying, as usual. There is a very limited market for this product, however, and it is not manufactured very extensively. The physiological characteristics are made use of in other compounds to a much greater extent, such as the following product.

16. Beef Peptone.—This is made by digesting beef by means of the pancreas of the beef. The tougher and leaner the beef, the better will be the yield of beef peptone. The meat is very finely minced, and to 25 parts of this are added 8 parts of the pancreas and 4 parts of water. The whole is allowed to digest in a jacketed kettle, at a temperature not exceeding 130° F., for 6 hours, after which time the solids will have all passed into solution through a true digestive process. During this time the mixture must be frequently stirred to facilitate the action. The solution is filtered through cotton-flannel bags after the manner of beef extract and the filtrate bleached in the same manner as pepsin solution, for which the same apparatus will serve.

The bleached solution is evaporated to dryness on a water bath or at a heat not exceeding 212° F. It may also, with economical effect, be evaporated to a thick consistency in a vacuum apparatus and afterwards dried. The yield of beef peptone from this process, using tough and lean meat, is 13 to 14 per cent. of the total weight of the meat and pancreas. This yield is rather high for the ordinary run of beef, from which a yield of about 10 per cent. could be obtained.

17. Pancreatin.—This is a digestive ferment existing in the fluids of the pancreas, or sweetbreads, of animals. Pancreatin is ordinarily made from the pancreas of the hog, in a similar or analogous manner to that of pepsin from hog stomachs. This product is not made to any extent in the average packing house, bordering, as it does, upon the field of the pharmacist.

The pancreatin solution, like the pepsin, is also very susceptible to decomposition, which may be and usually is prevented by the addition to the solution of an infinitesimal amount of thymol or chloroform, either of which does not interfere with the action of the ferment.

18. Rennet.—This is prepared from the fourth, or true, digestive stomach of the calf. It is not prepared in the packing house, the stomach being simply dried and sold in that condition.

ANIMAL EXTRACTS

19. General Remarks.—Animal extracts are made from the various organs of slaughtered animals, and are said to have great virtue in the treatment of diseases of man, in similar organs to those from which the various extracts are obtained. These extracts were first prepared by Dr. Hammond, of this country. The preparations are obtained from the healthy organs of the animals selected for this purpose. Each organ used is subjected to careful examination, in order to insure freedom from affections in any form.

Among these extracts are medulline, from the spinal cord of the bullock; cardine, from the heart of the bullock; testine, from the testes of the bull and the ram; ovarine, from the ovaries of the hog; musculine, from the muscular tissues of the bullock; thyroidine, from the thyroid glands of the sheep and the bullock; and cerebrin, from the brain of the bullock.

20. Process of Preparation of Animal Extracts. The original process as described by Dr. Hammond is given below:

“The process of preparation of the extract of these several organs, while individually somewhat different, does not materially vary from that used for the brain, which is as follows:

“The whole brain of the ox, after being thoroughly washed in water acidulated with boric acid, is cut into small pieces in a mincing machine. To 1,000 grams of this substance placed in a wide-mouthed glass-stoppered bottle, I add 3,000 cubic centimeters of a mixture consisting of 1,000 cubic centimeters each of saturated solution of boric acid in distilled water, pure glycerine, and absolute alcohol. This is allowed to stand in a cool place for at least 6 months, being well shaken or stirred two or three times a day. At the end of this time it is thrown upon a porous stone filter through which it percolates very slowly, requiring about 2 weeks for entirely passing through. The residue remaining upon the filter is then enclosed in several layers of aseptic gauze, and subjected to a pressure of over 1,000 pounds, the exudate being allowed to fall upon the filter and mixed with a sufficient quantity of the filtrate to cover it. When it has entirely filtered, it is thoroughly mixed with the first filtrate, and the process is complete.

“During the whole of this manipulation, the most rigid antiseptic precautions are taken. The vessels and instruments required are kept in boiling water for several minutes and are then washed with a saturated solution of boric acid. Bacteria do not form in this mixture under any circumstances, but it is necessary to examine it from time to time, microscopically, in order to see that no foreign bodies have accidentally entered. Occasionally, owing to causes that I have not determined, though I think it is due to variations in temperature, the liquid becomes slightly opalescent from the formation of a flocculent precipitate. It sometimes takes place in a portion of the extract kept under apparently identical conditions with other portions that remain perfectly clear. It can be entirely removed by filtration through Swedish filtering paper, previously sterilized, without the filtrate losing anything of its physiological or therapeutical power.”

The above process is the best that has so far been devised for obtaining these extracts, and when made, these conditions should be fully and most carefully maintained.

FERTILIZERS

FERTILIZER MATERIALS

21. Under fertilizer material is included practically all material that cannot be made use of in any other way. At one time the waste material of the packing house was not only a nuisance, but the disposal of it was a source of expense. Now, however, all refuse is utilized by making it into various grades of fertilizers, which are a source of income of no inconsiderable importance. The manufacture of fertilizers from the various waste materials will be taken up at length.

22. Tankage.—Tankage is the name properly applied to the sediment remaining in the tanks where meat scrap, with some bone, is rendered to separate the fat. The name is also applied to the refuse from tanking garbage, the dried product being known under the name *garbage tankage*. This product, however, is never made in packing houses. The name tankage is also loosely applied to mixtures that consist largely of bone and do not differ greatly in composition from pure bone.

After material has been cooked in the rendering tanks the fat is withdrawn from it, and when all that can be taken from the tank has been obtained, the cooked, hot material is allowed to drop into a box or vat in front and underneath the tank. Here the material is kept hot by the steam pipes in the vat, and whatever grease or fat rises to the surface is skimmed off. The latter is technically known as *skimmings*, and is recooked with the next charge of fresh material.

The water with which the material in the tank has been cooked becomes impregnated with more or less fat and also with a very large quantity of nitrogenous material. It is technically known as tank water and is the source of *concentrated tankage*, or *stick*. The treatment of tank water will be described later under concentrated tankage.

The material in the vat, after skimming, is drained free from the tank water and while still hot is placed in the

tankage or fertilizer press. This is ordinarily done by hand labor, but in the modern establishments the tankage is flowed from above into the press cloth on the forms that are placed on the platform of the press.

23. Pressing Tankage.—There are several forms of fertilizer presses in use. Among these are the knuckle-joint, the power-screw, and the hydraulic press. Fig. 1 shows the

FIG. 1

power-screw press, which is the style of fertilizer press preferred by many of the leading packers, and only by the use of this have they been able to handle the immense amount of material that is now utilized and made into a valuable fertilizer. It has the advantage of giving the same pressure at every point, thus pressing a small amount of material as well as large, without the handling of any blocking. It is made very heavy and strong, to stand rough usage, and

will give an immense pressure. Although shown with a double platform, it may be fitted with any other style of platform to suit the situation.

The cloth used for pressing tankage is made especially for this purpose. It is known as tankage press cloth, and is made of rather coarsely but strongly woven jute. A rack, Fig. 2 (a), is first placed on the platform *a* of the press. This rack is square, and made of wooden strips $\frac{3}{4}$ inch thick by $\frac{7}{8}$ or $1\frac{1}{8}$ inches wide, placed $\frac{1}{4}$ inch apart, with five or more elm strips 2 inches wide and $\frac{3}{4}$ inch thick nailed across, as

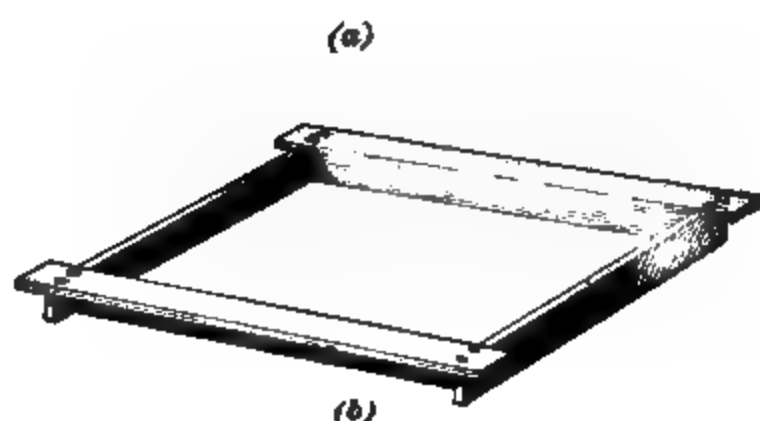


FIG. 2

shown. Wrought-iron nails of sufficient length to securely clinch in the elm are used.

The platforms *a* and *b*, running on a track, may be filled at any convenient place and afterwards run to the press. On the rack is a form as shown in Fig. 2 (b). This form is square inside and $3\frac{1}{2}$ inches deep. It is made by nailing together boards, 1 inch thick by $3\frac{1}{2}$ inches wide, in the form of the sides of a box. A board is nailed across each end, as shown, to serve as a guide and to give stiffness. Over this form is spread a cloth that is filled with tankage until the material is even with the top of the form. The

cloths should be sufficiently large to enable the sides and ends to be folded over, thus completely covering the material. The form is then raised and another rack placed on the layer thus made, the form being placed on this new rack, a cloth again placed over it, and another layer of wet tankage put in as before. From eight to twelve racks are used in one load and as many cloths, less one. When the last layer is made, the form is taken off and a rack used for the top of the load. The platform is then run on the track *e* to the press and the pressing begun. By placing the racks alternately across and lengthwise of the platform, the built-up load will be less liable to move or cant over and cause the rack to spread. A guide should be used in building the layers, so as to always have the form directly over the last layer. The load consisting of the different layers of material to be pressed is sometimes termed a *cheese*.

The power in pressing is applied by the pulleys *d*, which cause the gears to move the upper part of the press *c* downwards, thereby pressing out the water and grease remaining in the wet tankage. The racks afford channels for this water and grease to run out of each layer. By a reverse motion of the power, the upper part of the press is raised when the pressing is finished.

The advantage of the double platform is that while one load is being pressed on *a*, another may be built in the interval on *b*.

24. In Fig. 3 is shown the knuckle-joint fertilizer press with one load pressing and another load of material in layers, ready for running under the press when the first pressing is finished. The press is shown with double platform and power attachment, although they are made, if desired, with single platform and hand-power attachment. For small packing houses, this press has been found very satisfactory.

25. In using hydraulic presses for tankage, it is necessary to block the load by square pieces of wood between the

load of layers and the upright guides on the press. This press does excellent and rapid work, and presses the material very much drier than either of the presses illustrated.

The pressed material contains, approximately, 50 per cent. of moisture after removing from the press and shaking free from the cloths. The water and grease pressed out of the

FIG. 8

hot tankage run to a catch basin, where the grease, rising to the top, is skimmed off and recooked with refuse material for No. 2 tallow, or yellow grease.

26. Drying the Pressed Tankage.—The pressed material is now transferred to the drier for the final drying into a commercial product. Several forms of driers may be had.

Fig. 4 shows one of the most modern makes, which is

known as a single-cylinder drier. It is extremely simple in operation. The iron cylinder *A* is a jacketed or double shell in which are revolving arms, or paddles, operated by the shaft *b*. The fertilizer material to be dried is loaded into the cylinder through the door *c*, which is then closed, and the shaft is started revolving by the large cog wheel *f*, operated by the power applied to the pulley *e*. Steam under pressure circulates through the double shell, and the heat from it drives off the moisture in the material. The odors and gases coming from the fertilizer material pass through the pipes to the condenser, which is attached to all driers. The condenser has jets

of water running into it, which, coming in contact with

the hot gases, cool them, and at the same time cause the greater part of them to pass into solution with the water. The cylinders of the drier vary from 3 to 5 feet in diameter, and are from 10 to 16 feet long. The clutch *g* serves to start and stop the power that operates the drier.

The tankage is dried so that it contains 10 per cent. or less of moisture, which requires from 3 to 6 hours, depending on the class and quantity of material worked on. It is then allowed to drop out through the doors *d*. The material is so hot that if it were piled up in this condition, it would not become cold and would rapidly decompose. The hot material is spread on the floor about 3 inches deep. After the heat has gone from it the material is shoveled into piles; or if desired for immediate shipment, is put into second-hand bags, such as salt bags, which contain about 200 pounds each of the dried tankage.

27. Grading of Tankage.—Tankage is divided into several grades, which are quoted by the percentages of ammonia and phosphoric acid shown by analysis. For example, a 7-30 tankage would be one that analyzes 7 per cent. of ammonia and 30 per cent. of phosphoric acid. A low-grade tankage contains less than 10 per cent. of ammonia and a high percentage of bone phosphate, while high-grade tankage always contains 10 per cent. or more ammonia, and correspondingly less bone phosphate. Bone tankage, as the name implies, is that containing mostly bone. Tankage is also classed as *unground*, *crushed*, and *ground tankage*. The former is tankage that is dried without subsequent grinding or crushing of large sinews, meaty pieces, or bones. Crushed tankage consists of unground tankage broken up sufficiently to pass through a screen of about 1- or 1½-inch mesh. Ground tankage is tankage ground to fine condition. This material will pass through a sieve of about ½-inch mesh. The usual difference in trade values of each is \$1 per ton, ground tankage being the most valuable.

28. Concentrated Tankage.—This is also known under the trade name *stick*. It is made by evaporating the tank

water from the cooking of meaty material. Tank water contains a considerable amount of dissolved nitrogenous matter, which, when recovered in the form of concentrated tankage, furnishes a valuable fertilizer material.

The tank water is run to receivers and thence to evaporators, and there evaporated to a sirupy consistency. The tank water, previous to evaporation, has mixed with it some form of cheap sulphates, such as copperas (sulphate of iron). The proportions used are 500 pounds of copperas to each 2,000 pounds of dry substance in the tank water. The mixture is concentrated to about 30 per cent. of moisture, when it is placed about 1 inch deep in sheet-iron pans 30 inches long, 15 inches wide, and 3 inches high. These are placed in a drying oven and baked to dryness. The material is then knocked from the pans and ground fine. The product resembles ground blood and will analyze about 15 to 16 per cent. ammonia on a dry basis. This material, like dried blood, is sold by the "unit" value, being quoted commercially at a certain price per unit. The unit is but an arbitrary commercial standard, the chemical percentage of ammonia being taken. Each per cent. of ammonia equals one unit per ton.

When large quantities are treated, the form of evaporator used is the vacuum pan, which evaporates large quantities in an economical manner. Where small quantities are to be evaporated, the roller evaporator, similar to Fig. 6, *Packing-House Industries*, Part 2, may be used with good effect.

It is very essential that some efflorescing material, as copperas, be used in making concentrated tankage, as without this it would be impossible to evaporate the tank water to dryness. Even if the latter condition could be obtained, the material alone, being very hygroscopic, would absorb moisture so rapidly as to become sticky, lumpy, and, consequently, useless as fertilizer material.

29. Dried Blood.—The liquid blood is conducted from the slaughtered animals into a tank, or vat, where it is boiled with open steam for 20 minutes. This coagulates the

albuminous matters and renders them practically insoluble. The clotted blood is then pressed in precisely the same manner as tankage. The pressed-out water has no value and is allowed to waste. The cloth for pressing blood is of closer texture but of the same material as tankage cloth.

The pressed blood from the fertilizer press contains about 50 per cent. of moisture. It is broken into small lumps and passed through a disintegrator, which tears it into lumps about the size of a bean. This is then passed to the drier, from which it emerges as dried blood, containing, however, numerous hard lumps that are technically known as *blood screenings*. The blood is screened through a sieve, and the screenings are ground by themselves to the required degree of fineness and mixed with the screened blood. Instead of sifting the dried blood as it comes from the drier, it may all be put through the mill and ground fine. The hot material is spread on the floor in a thin layer, to allow the heat to escape. It is then made into a pile or sacked like tankage.

Ground blood is always sold on the unit basis of contained ammonia, the percentage of which will vary according to its freedom from extraneous material and moisture. Clean ground blood should contain from 16 to 17 per cent. of ammonia and from 2 to 3 per cent. of moisture. When mixed with refuse, it may not contain over 9 per cent. of ammonia.

30. Raw Bone and Raw-Bone Meal.—The bones that have not been subjected to pressure in cooking, such as shin, knuckle, jaw, and head bones, furnish raw bone. After these have been freed from as much grease as possible, and dried, they are ground in a bone mill to fineness,

making raw-bone meal. This mill is sometimes termed a disintegrator. Fig. 5 shows the disintegrator closed and Fig. 6 shows the same open. A peculiarity of the bone mill is that the material is ground by impact upon rounded iron bars and upon itself. The open machine shows the revolving barred wheels. The inside wheel *a* revolves in the opposite direction from the outside one *b*, both revolving at a very high rate of speed. As the bones are fed into the hopper; the revolving wheels keep them flying around until of a fine, dusty condi-

FIG. 6

tion, when they pass through the bottom of the mill. The material is screened and any pieces of bone that have escaped grinding are returned to the mill. This, however, is seldom necessary.

After cooking, raw bone contains so much of the original substance that it is always necessary, previous to grinding, to crush the bone by means of a bone crusher, which is built on a plan similar to the ordinary rock or ore crusher. Raw-bone meal as produced in the packing house will contain from 4 to 5½ per cent. of ammonia and from 20 to 25 per cent. of phosphoric acid.

31. Steamed Bone and Ground Steamed Bone.

These materials are made from bones that have been cooked under pressure. All bones that are not useful nor desirable for other purposes are made into steamed bone. They comprise rib bones, large knuckles, back bones, etc. From these bones are produced *butter-stock tallow* and *glue liquors*. For producing the latter, in connection with steamed bone,

special cooking of the bones is necessary, which is not essential when they are simply cooked for the tallow and residual bone.

32. Cooking Bones for the Recovery of Tallow and Steamed Bone.—The bones are put into the rendering tank and cooked for 7 to 10 hours at about 40 pounds steam pressure per square inch, which effectually removes the tallow from the bones. After a period of rest, to allow the tallow to rise, the latter is drawn off and the bones and water are dumped into a vat, from which any floating tallow remaining is removed. The water is drained away, as it seldom contains sufficient nitrogenous material to warrant the expense of evaporation for concentrated tankage. The cooked bones are very friable and porous. After draining on the floor for several hours, they are removed and made into piles. The heat generated in these piles is amply sufficient to dry the bones without passing them through a drier. They may also be dried by simply spreading in the open air.

33. Cooking Bones for the Recovery of Tallow, Glue Liquors, and Steamed Bone.—The method of cooking bones for the production of glue liquors, together with the tallow, is carried out as follows:

The bones, which are loaded into the tank, are allowed to stand in water at about 130° F. for 1 hour. This water is run off and, where beef extract is made, is used for this purpose. The bones are again covered with water and cooked for 6 hours with the head out of the tank. This is known as *open cooking*. The material is allowed to settle, and the resultant butter-stock tallow is drawn off. The head is now put into the tank and the bones are cooked for 2 hours under 30 pounds pressure per square inch. The glue liquor after settling is drawn off, more water is put on the bones, and a further pressure cooking of 3 hours given, after which the tallow and glue liquor are drawn off. The material is now cooked at 40 pounds pressure per square

inch for a few hours, when, after drawing off the tallow, the cooked bones are dumped from the tank into the vat underneath and treated as just described.

The following are the results of tests on the boiling of rib bones. In test No. 1 the bones were boiled for the recovery of bone and tallow, and in test No. 2 the same material was boiled for the recovery of bone, tallow, and glue.

Test	Dry Glue. Per Cent.	Tallow. Per Cent.	Dry, Steamed Bone. Per Cent.
No. 1.....	11.50	7.48	40.80
No. 2.....	21.27	10.88	31.64

Analyses of the dry, steamed bone obtained above gave the following partial compositions:

Test	Ammonia. Per Cent.	Bone Phosphate. Per Cent.	Fat. Per Cent.
No. 1.....	3.66	59.21	7.67
No. 2.....	1.45	72.01	2.33

While the cooking for glue by this method consumes more time and is more laborious, the increased yield in the tallow obtained, together with the value of the glue liquor, makes it a more profitable method than cooking the bones for tallow and bones only.

The average steamed bone will analyze from 2 to 3 per cent. ammonia and about 60 per cent. bone phosphate. The dried, steamed bone is made into steamed-bone meal by grinding through the ordinary bone mill. For this purpose, they need not be previously crushed, as their brittle nature makes them very easily disintegrated.

34. Azotine.—This material is made from the residue of cooked cracklings, which, after being subjected to high pressure to extract all the grease possible, is passed through a drier and then ground fine. The material will shrink in weight from 35 to 40 per cent. from the raw to the finished material, and loses at the same time several per cent. of ammonia. This material is not made in the large packing houses, but is derived mostly from small tallow and grease renders. Azotine is sold on the unit basis of contained ammonia, that made from pork material containing about 2 per cent. more ammonia than that made from beef. Commercial azotine will analyze about 15 per cent. ammonia on a basis of 10 per cent. moisture.

35. Hoof Meal.—This is made from cattle hoofs. The latter are cooked in the pressure tank for 5 hours at about 40 pounds steam pressure per square inch, when they are dried thoroughly. This must be the case, for if they contain any appreciable moisture, it will be impossible to grind them. The dried hoofs are ground in the regulation bone-grinding mill to the fineness of bone meal. With the hoofs may also be placed useless horns, which when ground also furnish hoof meal. This material has a high content of nitrogen, analyzing on a dry basis over 19 per cent. of ammonia. An analysis of hoof meal gave 2.1 per cent. moisture and 19.05 per cent. ammonia.

MIXED FERTILIZERS

36. General Remarks.—For mixing fertilizers to make complete fertilizers, it is necessary to add to the bones and tankage other materials as diluents, and also some form of potash. The latter is most commonly commercial potassium chloride and at times potassium sulphate. The sulphate is regarded as preferable, especially for tobacco fertilizers. The diluents, or fillers, may be any cheap material, such as ashes ground fine or earth.

Complete fertilizers are materials that contain the elements necessary for the growth of organic matter which are first extracted from the soil by growing crops. They comprise nitrogen (ammonia), phosphoric acid, and potash.

Direct fertilizers contain certain forms of plant food that contribute directly to the growth and substance of plants. Such materials may contain nitrogen, potash, or phosphoric-acid compounds or any two, or all three, of these forms of plant food.

Indirect fertilizers are those that do not in themselves furnish directly to the soil any needed plant food, but whose chief value depends on the power they possess of changing unavailable forms of plant food into available forms. Indirect fertilizers include lime, gypsum, and salt.

37. Ingredients of Mixed Fertilizers.—Among the various and most common ingredients used in making complete fertilizers are dried blood, tankage, azotine, bones, potassium chloride, potassium sulphate, sodium nitrate, ammonium sulphate, and various forms of phosphates derived principally from phosphate rock.

Tankage, containing, as it ordinarily does, from 7 to 10 per cent. of ammonia, is in too concentrated a form to be used by itself as a fertilizer and is, at the same time, too expensive. Although, as a rule, there is nothing objectionable in its use in its natural state, it lacks the usual requirements in a commercial fertilizer of the very essential ingredient potash.

Bone, in the form of raw-bone meal, is frequently used for a direct fertilizer. But the objection to its use is its slow availability by the soils and the insoluble form of most of the phosphoric acid it contains.

A source of nitrogen frequently used in complete fertilizers is found in sodium nitrate, or Chile saltpeter, which contains about 15½ to 16 per cent. of nitrogen. This is ordinarily used in connection with the phosphates, tankage, and other nitrogenous materials, such as bones. The disadvantage of using sodium nitrate is its free solubility in water, thereby

washing out of the soil too readily. It is very adaptable, however, for crops that mature in a short growing period.

Phosphate rock is a mineral phosphate found in various states. In a raw condition it contains from 25 to 35 per cent. of phosphoric acid, which, however, is in an insoluble condition and, consequently, must be treated with sulphuric acid before the phosphates can be made available. The quantity of acid required varies with the amount of phosphates present, which has to be determined by analysis in the usual way. (See *Quantitative Analysis*.)

Acid phosphates are known under various names, such as superphosphates, dissolved rock, etc. They are also formed by treating bones, bone black, bone ash, etc. with sulphuric acid. By such treatment there are formed soluble phosphates of calcium and calcium sulphate (gypsum) in nearly equal proportions. All forms of bone products are valuable fertilizers, as they furnish phosphoric acid and ammonia also.

Ammonium sulphate is a by-product from the manufacture of illuminating gas or coke. There are occasions, however, when its high price prevents its use in commercial fertilizers.

Potassium salts used in the manufacture of fertilizers are obtained principally from the Stassfurt mines. The potassium chloride generally contains from 50 to 53 per cent. of actual potash. Potassium sulphate from this source ordinarily contains from 48 to 51 per cent. of actual potash.

38. Mixing the Ingredients.—The raw materials above described are mixed in various proportions, dependent on the use of the fertilizer for certain crops and also on the sale price of the finished product. The materials should all be in a finely ground condition, as the mechanical condition of a fertilizer is an important consideration, in addition to its chemical composition, the degree of pulverization controlling the rate of solubility of the different ingredients.

The mixing of the various ingredients is merely a mechanical operation. It is most commonly done with some form of fertilizer mixer. It is very essential that the several

ingredients should be thoroughly incorporated with one another. The most common form is that of a shallow, revolving pan, in which is placed apparatus for mixing the fertilizer while the pan is revolving. Any form of mixer that will fill the requirements of thorough incorporation is adapted to this purpose. With small quantities, it has been accomplished by numerous siftings and subsequent hand mixing, which method is serviceable for experimental laboratory mixtures. Mechanical mixers are built that are capable of mixing from 10 to 150 tons of fertilizer every 10 hours.

39. Formulas for Fertilizer Mixtures.—Each manufacturer has his own formula by which his fertilizer is made. A few formulas will suffice to show the general method of combining the ingredients. For a fertilizer adapted for wheat and grass, the following mixture is made:

Steamed bone.....	1,200 lb.
Potassium chloride	45 lb.
Filler	755 lb.
Total	2,000 lb.

An analysis of this mixture will be about as follows:

Ammonia	2 to 2.5%
Available phosphoric acid.....	8 to 10.0%
Potash.....	1 to 2.0%

Another mixture is as follows:

Steamed bone.....	900 lb.
Tankage (low grade).....	190 lb.
Potassium chloride	210 lb.
Filler	700 lb.
Total	2,000 lb.

An analysis of this is about as follows:

Ammonia.....	2 to 3%
Available phosphoric acid.....	8 to 9%
Potash.....	5 to 6%

A mixture made for a potato fertilizer is compounded with the following ingredients:

Steamed bone.....	1,000 lb.
Sodium nitrate.....	212 lb.
Tankage (low grade).....	150 lb.
Potassium chloride.....	365 lb.
Filler	<u>273 lb.</u>
Total	2,000 lb.

This mixture will analyze as follows:

Ammonia.....	4 to 5%
Available phosphoric acid	7 to 8%
Potash.....	9 to 10%

A so-called "guano" is made from the following formula:

Steamed bone.....	570 lb.
Sodium nitrate.....	200 lb.
Tankage.....	900 lb.
Potassium chloride.....	<u>330 lb.</u>
Total.....	2,000 lb.

This mixture makes a very high-grade fertilizer, containing no filler. It analyzes about as follows:

Ammonia.....	6 to 7%
Available phosphoric acid.....	5 to 6%
Potash.....	8 to 9%

A mixture composed of

Bone meal.....	400 lb.
Dissolved bone black.....	400 lb.
Dried blood.....	300 lb.
Sodium nitrate.....	200 lb.
Dissolved phosphate rock.....	350 lb.
Potassium chloride.....	<u>350 lb.</u>
Total.....	2,000 lb.

was found to give, upon analysis,

Nitrogen.....	4.09%
Available phosphoric acid.....	9.59%
Insoluble phosphoric acid.....	2.50%
Potash.....	9.62%

ANALYTICAL METHODS AND TESTS OF PRODUCTS

DETERMINATION OF GRADE OF OILS, TALLOWES, GREASES, ETC.

40. The following analyses and tests, together with those referred to in *Quantitative Analysis* on the analysis of fats and also those on the examination of fertilizers, are sufficient to give the student a knowledge of the most important methods used in packing-house laboratories.

41. Determination of Melting Point of Fats.—Many methods have been devised for ascertaining this, but there is none that has been found to give entirely satisfactory results in all cases. Among the best methods are those described by Wiley, in "Principles and Practice of Agricultural Analysis." The method adopted by the Official Agricultural Chemists will be described, together with a method modified by Hebner and Angell, which has been found to give very satisfactory results where a number of samples of the same class of material are to have their melting points determined. This method will be found particularly applicable to butters and butterines. The latter method is carried out in the following manner:

42. Sinking-Point Method of Hebner and Angell. A small pear-shaped float is blown from a piece of glass tubing and made about 1 centimeter in diameter and 2 centimeters long. The stem of the pear is drawn out and broken off, and while the bulb is still warm, the open end of the stem is held in mercury. A small quantity of this substance, sufficient in amount to cause the float to sink slowly through a melted fat, is introduced into the bulb of the apparatus and the stem sealed. The whole bulb should displace about 1 cubic centimeter of liquid and weigh, after filling with mercury, about 3.4 grams.

In conducting the experiment, about 30 grams of the dry, melted fat is placed in a large test tube and cooled by immersing the tube in water at a temperature of 15° C. The tube containing the solidified fat is placed in a bath of cold water and the sinker is placed in the center of the surface of the fat. The bath is then slowly heated until the float disappears. The temperature of the bath is read just as the bulb of the float disappears.

43. Spheroidal State Method.—This method for determining the melting point of fats has been adopted by the official chemists. It depends on the fact that a disk of fat when floating in suspension in a liquid assumes a spheroidal form when melted. The method is described by Wiley as follows:

In the preparation of the apparatus there are required (*a*) a piece of ice floating in distilled water that has been recently boiled and (*b*) a mixture of alcohol and water of the same specific gravity as the fat to be examined. This is prepared by boiling distilled water and 95-per-cent. alcohol for a few minutes, to remove the gases that they may hold in solution. While still hot, the water is poured into the test tube described below until it is almost half full. The test tube is then nearly filled with the hot alcohol, which is carefully poured down the side of the inclined tube to avoid too much mixing. If the alcohol is not added until the water has cooled, the mixture will contain so many air bubbles as to render it unfit for use. These bubbles will gather on the disk of fat as the temperature rises and finally force it to the top.

The apparatus for determining the melting point is shown in Fig. 7, and consists of an accurate thermometer *a*, for determining the melting point, reading easily tenths of a degree (it is advisable to use a cathetometer for reading the thermometer, but this may be done with an eye glass if held steadily and properly adjusted); a thermometer *c* for regulating the temperature of the bath; a tall beaker *d*, 35 centimeters high and 10 centimeters in diameter; a test

tube *e*, 30 centimeters long and $3\frac{1}{2}$ centimeters in diameter; a stand *f* for supporting the apparatus; some method of stirring the water in the beaker (for example, a blowing bulb *g* of rubber and a bent glass tube extending to near the bottom of the beaker).

The disks of fat are prepared as follows: The melted and filtered fat is allowed to fall from a dropping tube, from a height of about 20 centimeters, on a smooth piece of ice floating in recently boiled distilled water. The disks thus formed are from 1 to $1\frac{1}{2}$ centimeters in diameter and weigh about 200 milligrams. By pressing the ice under the water, the disks are made to float on the surface, from whence they are easily removed with a steel spatula, which should be cooled in the ice water before using. They should be prepared a day or at least a few hours before using.

FIG 7

The test tube containing the alcohol and water is placed in a tall beaker containing water and ice and left there until cold. The disk of fat is then dropped into the tube from the spatula, and at once sinks until it reaches a part of the tube where the density of the mixture of alcohol and water is exactly equivalent to its own. Here it remains at rest and free from the action of any force save that inherent in its own molecules.

The delicate thermometer α is placed in the test tube and lowered until the bulb is just above the disk. In order to secure an even temperature in all parts of the alcohol mixture, in the vicinity of the disk, the thermometer is gently moved from time to time in a circularly pendulous manner.

The disk having been placed in position, the water in the beaker is slowly heated and kept constantly stirred by means of the blowing apparatus g already described. When the temperature of the alcohol-water mixture rises to about 6° below the melting point, the disk of fat begins to shrivel and it gradually rolls up into an irregular mass.

The thermometer is now lowered until the fat particle is even with the center of the bulb. The bulb of the thermometer should be small, so as to indicate only the temperature of the mixture near the fat. A gentle rotatory movement from time to time should be given to the thermometer bulb. The rise of temperature should be so regulated that the last 2° of increment require about 10 minutes. The mass of fat gradually approaches the form of a sphere, and when it is sensibly so, the reading of the thermometer is to be made. As soon as the temperature is taken, the test tube is removed from the bath and again placed in the cooler. A second tube containing alcohol and water is at once placed in the bath. The test tube (ice water having been used as a cooler) is of low enough temperature to cool the bath sufficiently after the first determination, which should be only a trial, and the temperature of the bath should be so regulated as to reach a maximum of about 1.5° above the melting point of the fat under examination.

The edge of the disk should not be allowed to touch the sides of the tube. This accident rarely happens; but in case it should take place and the disk adheres to the sides of the tube, a new trial should be made.

Triplicate determinations should be made and the second and third results should show a near agreement.

In this connection, with the determination of melting points, the student is referred to the melting point of waxes in *Quantitative Analysis*. The methods there described are

also applicable in some cases to fats, but the method just described has met with much success and approval. For rapid determinations with a large number of samples, that of Hebner and Angell is to be preferred.

44. Determination of Tallow in Lard.—It is frequently of importance to have a rapid method of ascertaining whether or not a lard is adulterated with beef fats. While a large proportion of the latter would to the practical chemist be made evident by the altered physical characteristics of pure lard, yet an admixture of a small percentage of tallow or oleo stearin would easily pass unnoticed. ✓

45. Wesson's Method for Tallow in Lard.—David Wesson has devised a ready method of determining small quantities of tallow, which, while not exact in the scientific sense, is so eminently practical, rapid, and easily applicable in the packing house that it is relied on to give approximate results. It is obvious that the results obtained by this method must be confirmed by further investigation, iodine, number, etc., where the result is of importance in legal cases, disputes, etc., but for the matching or duplicating of a lard for trade purposes, the method has great value and utility.

All fats in a state of fusion, a little above their melting points, may be considered as solutions of their solid glycerides in the liquid oils or the oleins. For example, cottonseed oil at ordinary temperatures contains from 20 to 25 per cent. of solid glycerides, which crystallize out as stearin. When the oil is chilled and at a temperature of 0° C., the oil becomes a solid fat. In like manner, prime steam lard at 32° C. is a clear oil from which its solid glycerides crystallize on cooling, until at 25° C. the lard becomes a solid fat. Prime steam lard contains about 35 per cent. of solid glycerides and about 65 per cent. of olein, which holds them in solution at 30° to 32° C., and in some instances, lower.

The solid glycerides from tallow are far less soluble in olein than those from lard, and crystallize out at much higher temperatures. Owing to this fact, it is possible to detect with ease and certainty the solid glycerides of beef

fat, namely, oleo stearin, when it exists in lard in quantities of 5 per cent. and upwards.

46. The process, which is very simple, is conducted as follows:

First, determine the titer of the fat under examination. The Dalican titer or crystallization temperature of the fatty acids indicates, approximately, the proportions of liquid and fluid acids present.

Second, prepare with materials of known purity prime lard and prime-lard stearin, a mixture that will possess about the same titer as the sample. Pour some of the melted standard into a test tube, and a like quantity into another; at the same time have another test tube containing a mixture of lard hardened to the same titer with oleo stearin. The tubes are all brought to the same temperature in a beaker of hot water and immersed in a bath of water at about 35° C. As the bath cools down, the tubes are watched from time to time, and the temperatures and times noted at which they deposit crystals and their manners of crystallization.

If the sample under examination consists of pure lard stock, it will cool in a similar manner to the standard mixture. If oleo stearin is present, the sample will deposit crystals, or become cloudy sooner and at a higher temperature than the standard, and comparison with the samples prepared with oleo stearin will show the approximate percentage, which will be confirmed by the Dalican titer, taking into consideration the approximate amount of cottonseed oil that may be present. The manner of cooling is quite characteristic. Pure lard stock generally forms comparatively large, stellate, or starlike bundles of crystals, while those from oleo stearin are very small, and appear as a cloudiness. A few trials with mixtures of known composition will make these appearances familiar and unmistakable.

It is of the utmost importance that the lards should be dry and clear, as moisture will cause a lard to cloud above

its normal temperature. With this test, soft tallow and oleomargarine, having not too low melting and cooling points, on account of the preponderance of olein in them, give the indications of quantities of oleo stearin proportional to their hardness, and in some cases where the flavor and appearance of the sample indicate tallow, rather than oleo stearin, it is the custom to report tallow equivalent to so much oleo stearin.

With lards of comparatively high titer and consequently high cooling point, it is necessary to consider the effect of lard stearin on the titer when calculating the probable composition. Ten per cent. of oleo stearin mixed with pure lard will deposit crystals and show cloudiness before a mixture of 50 per cent. of lard and 50 per cent. of lard stearin.

47. The arrangement of the apparatus used for this determination is shown in Fig. 8. A beaker *a* of about 750 cubic centimeters capacity is covered with a piece of thin wood, cardboard, or metal *c*, provided with a circular aperture, through which passes a smaller beaker *b*. The cover *c'* is provided with orifices for a number of test tubes *l*, which contain the samples and standards of comparison. A thermometer *t* indicates the temperature in *b*. The inner beaker *b* is filled with water up to *w*, which should be a little above *w'*, the surface of the melted lards.

FIG. 8

48. Determination of Unsaponifiable Matter in Oils, Etc.—Ten grams of lard or oil is accurately weighed off in a porcelain dish and saponified with alcoholic potash. The solution is evaporated on a water bath to dryness, moistened when dry with methyl alcohol, and again evaporated to dryness. The soap is dissolved to a clear solution with distilled water and this solution shaken out in a separatory funnel with light boiling gasoline or rhigolene. The gasoline is retained in the separatory funnel by running off the watery underlying solution.

The gasoline remaining in the funnel is washed several times with water to remove any dissolved soap, and finally withdrawn into a weighed cup or flask. The gasoline is either distilled or evaporated off and the receptacle and contents weighed, after drying for 2 hours at 100° C. The increase in weight divided by the amount of substance taken will give the percentage of unsaponified matter present in the oil or fat. In connection with this determination, the student should refer to *Quantitative Analysis*. The method there described combined or used in connection with the above will enable him to detect adulteration of animal oils with mineral oils.

49. Cold Test of Animal Oils.—This is a trade requirement with most animal oils. It is made as follows: The sample of oil must be thoroughly mixed, to insure uniformity. If the oil is frozen or chilled, it must be warmed and melted to a uniform consistency.

About 1 ounce of the oil to be tested is put into a common 4-ounce sample bottle, and in it is placed a short, stout thermometer. The bottle is then placed in a situation where the oil will become frozen; if necessary, a freezing mixture is used. (In connection with this test, see the "Cloud Test in Mineral Oils," *Quantitative Analysis*.) When the liquid has become solid throughout, the bottle is removed from the cold and the oil allowed to soften, at the same time thoroughly stirring and mixing it by means of the thermometer, until the mass will run from one end of the bottle

to the other. The bottle is now grasped by the neck by one hand, having in the same hand a little waste or a towel, which encloses the thermometer. The latter is withdrawn through the waste or towel, to wipe far enough to see the mercury, and the temperature is observed. The reading is the cold test of the oil.

50. Determination of Free Fatty Acids in Oils, Tallow, Etc.—A supply of neutralized alcohol is made by dissolving in ordinary alcohol a small quantity of phenolphthalein and cautiously adding, drop by drop, a weak solution of alkali until, after persistent shaking, the alcohol retains a faint pink color. All free acid is now neutralized.

A known weight of oil or fat is placed in an ordinary 4-ounce sample bottle, and to it is added 50 or 60 cubic centimeters of the neutralized alcohol and the whole shaken and heated on the water bath for 15 to 20 minutes, or until hot. If the sample is wholly free from acid, the pink color of the alcohol remains unchanged. If free acid be present, the alcohol is decolorized. A seminormal solution of caustic soda is now carefully added until, after successive shakings, the pink color remains to the alcoholic solution. The reaction is very sharp, and the end point, with all material except very dark oils or greases, well defined. When testing the latter, a much larger quantity of alcohol is taken, and the solution after each shaking allowed to settle, by which the color of the underlying solution may more easily be observed.

51. In ordinary practice, it is customary to consider the free acid present in oils and fats as oleic acid, although this may not actually be the case. The molecular weights of oleic and stearic acids are not so widely different that the result is materially affected. For example, 1 cubic centimeter of seminormal caustic solution corresponds to .142 gram of stearic acid and to .141 gram of oleic acid.

For oils of average grade, 10 grams is usually taken for the test. For material containing very little free acid, the quantities taken for the test must be correspondingly

increased to obtain an accurate result. For poor tallows, from 20 to 25 grams is generally taken. For good tallows and oleo stearins, 50 grams is taken for the test.

52. Detection of Cottonseed Oil in Oil and Lard. Many methods are in use for the purpose of detecting adulterations of various products with cottonseed oil. While it is not advisable to put entire reliance on any one test, that of Millian has been used with good results in the packing-house laboratory. The method depends on the darkening of the fatty acids of cottonseed oil by the action of nitrate of silver upon them. The fatty acids of pure lard or olive oil are not discolored by the action of nitrate of silver. The process as performed by Millian follows:

In a porcelain dish of 250 cubic centimeters capacity, 15 cubic centimeters of the substance to be tested for the presence of cottonseed oil is heated to 110° C. Then, upon the oil is poured a mixture of 10 cubic centimeters of sodium hydrate, 36° Baumé, with 10 cubic centimeters of 90-per-cent. alcohol. While the heated mass is still limpid and homogeneous, 150 cubic centimeters of hot distilled water is added and the mixture heated to drive off the alcohol. This requires but a short time. The solution is now decomposed with $\frac{1}{10}$ normal sulphuric acid to acid reaction. The fatty acids are immediately removed with a platinum spatula and washed two or three times with cold distilled water. They are then placed in a test tube ($2\frac{1}{2}$ centimeters by 9 centimeters), and 15 cubic centimeters of 92-per-cent. alcohol and 2 cubic centimeters of a 3-per-cent. solution of nitrate of silver added.

The test tube with contents is placed in a water bath at 90° C. and about one-third of the alcohol evaporated, when 10 cubic centimeters of distilled water is added, the heating continued a few minutes longer, and the coloration of the fatty acids noted. In the absence of cottonseed oil they remain perfectly white; while if cottonseed oil be present, the depth of color depends on the amount of cottonseed oil present in the substance.

In connection with this test, the student should also apply those described in *Quantitative Analysis*. When any single test fails to give results of a most decisive character regarding adulteration, all tests applicable should be applied to the lard, fat, or oil under consideration. Taking all the results together, the absence or presence of cottonseed oil or other adulteration may be determined with comparative certainty.

53. Examination for Impurities in Solid Fats, Oils, Etc.—This subject is covered fully in *Quantitative Analysis*, and further methods under the examination of waxes, also treated there, may be applied to these substances. It may be said that if one test is to be preferred over another for an examination of purity in lard, it should be that of the Hübl iodine number, which is carried out as directed in *Quantitative Analysis*.

54. Dalican Test for Determining the Titer of Tallow and Stearin.—The determination of the titer of a fat is to ascertain the temperature, expressed in degrees centigrade, at which the free fatty acids, extracted from a fat, solidify. This test is also called the *hardness*, or *freezing-point, test*. Animal fats consist largely of the glycerides of oleic, stearic, and palmitic acids. Almost every animal fat contains a small percentage of free fatty acid, together with a smaller or larger percentage of impurities.

The determination of the solidifying temperature of the fatty acids of a given fat requires, first of all, that the fatty acids be extracted from the fat, and separated from the glycerine, combined with which they form, as glycerides, the bulk of the fat. In order to obtain the free fatty acids, it is necessary to follow a roundabout way, as it is impossible to separate the fatty acids from the glycerine directly, except by subjecting the fat to a degree of heat and pressure and the action of a relatively smaller quantity of chemicals, which are apt to cause changes in the free fatty acids.

When an animal fat is treated and heated with a sufficient quantity of some strong alkali, such as caustic soda or caustic

potash, the glycerides are decomposed; the alkali unites with the free fatty acids, thereby forming soap and liberating the glycerine. The soap formed, when treated with a stronger mineral acid, such as hydrochloric or sulphuric acid, is decomposed. The hydrochloric or sulphuric acid, combining with the alkali of the soap, leaves the free fatty acid as an oily layer floating on top of the hot aqueous solution of the other constituents. To separate this oily layer, the aqueous solution may be drawn off from beneath the fatty acid or the entire mass may be cooled, when the fatty acids will congeal, forming a solid cake, which can be removed from the aqueous solution. By washing the free fatty acids with distilled water, the adhering traces of mineral acid, etc. are removed; by a subsequent drying, all the adhering water is removed, and the fatty acids obtained free from any of the substances originally combined with or added to them in the course of separation.

55. It is absolutely necessary to extract all the fatty acids from the fat and to have no fatty acids in combination with glycerine, left in the free fatty acids, the solidifying temperature of which is to be determined. In order to be sure that no such glycerides remain mixed with the fatty acids, it is necessary to saponify the fat completely, that is, to unite all the fatty acids existing in the fat with the alkali. To insure and facilitate the completeness of saponification, alcohol is added when the fat is heated with the strong caustic alkali. The presence of alcohol accelerates the action of the caustic, and under proper conditions insures the complete saponification. The alcohol may, however, cause an error if it is not completely removed by evaporation before the soap formed is decomposed by mineral acid. If alcohol is present in the soap when the mineral acid is added, the free fatty acid may combine with such alcohol and form a substance that, mixed with the free fatty acid, will reduce the solidifying temperature of the fatty acid.

Any glycerides left in the fatty acids will also have the same effect. It is therefore absolutely necessary to obtain

complete saponification and to drive off all the alcohol from the soap formed before such soap is decomposed by a stronger acid. When, with the exception of water, the fatty acids are separated from all the other substances, there only remains the freeing of the fatty acids of such water before they can be tested as to their solidifying point. It has been demonstrated that the manner in which this water is removed may materially influence the solidifying point. The titer is found $.5^{\circ}$ higher when the fatty acids are previously heated for 2 hours at 100° C. than when such heating is not done, or done only for a materially shorter time.

Bulletin No. 13 of the United States Department of Agriculture, Division of Chemistry, 1889, gives the official method of the Dalican test as practiced by the government chemists, but while it gives minute details for ascertaining the solidifying point of the fatty acids, it fails to give directions as to the manner of separating the fatty acids from the fat. To this lack of definite directions may be attributed some of the differences in results obtained by different chemists.

56. For the preparation of the soap and thereafter the free fatty acids from tallow, etc., the original Dalican saponification method is usually preferred. Dalican proposed to heat 50 grams of tallow, etc. to a temperature of 200° C., and to pour into the hot tallow a mixture of 40 cubic centimeters of caustic soda, specific gravity 1.33, and 33 cubic centimeters of 95-per-cent. alcohol, while constantly shaking or stirring the mass. This operation is usually carried on in an enameled iron dish. In heating the oil in such a dish to 200° C., a sufficient amount of heat is stored in the dish to drive off all or most of the alcohol and to produce an almost dry, crumbly soap, which is then dissolved in boiling water and decomposed by mineral acid.

For this purpose, 150 cubic centimeters of dilute sulphuric acid (sp. gr. 1.143) will be sufficient. The fatty acids rise to the top of the liquid in pasty, flocculent masses. The

liberated fatty acids are then boiled until they appear on the top of the liquid as a clear, oily layer. The entire mass is then transferred to a separatory funnel. The aqueous underlying solution is next drawn off and the fatty acids washed with very dilute, hot sulphuric-acid solution, and then with hot water, until the wash water gives no acid reaction. The fatty acids are then transferred to a beaker and dried for 2 hours at 100° C.

In the determination proper, the following apparatus is employed: A thin-walled test tube (1.4 inches by 6 inches) is fixed by means of a cork in a suitable bottle. A centigrade thermometer, reading from 1° to 60° and graduated in $\frac{1}{10}$ degrees, is fixed in the second test tube by a second cork, which must be sufficiently loose to permit of an easy stirring of the contents of the tube with the thermometer. If the stem of the thermometer be shortened in the making of it, by having an enlargement blown in the bore between 1° and 25°, the amount of mercury above the surface of the fatty acids is thus diminished and a very appreciable error consequently avoided.

The test tube is filled with the decanted and dry fatty acids from the beaker to within $\frac{1}{4}$ or $\frac{3}{4}$ inch from the top. The thermometer is inserted into the liquid to about the 35° mark and the liquid fatty acids stirred until they become quite opaque and partial solidification sets in. The thermometer should clear the bottom of the tube by about $1\frac{1}{4}$ or 2 inches, the tube inside the bottom being protected from cold surfaces, on which it may rest by a wad of cotton placed in the bottom of the bottle.

After stirring rapidly in a circle ten or twelve times, the thermometer is allowed to stand at rest. The mercury now begins to rise in consequence of the latent heat liberated from the solidifying fatty acids; the highest temperature noted is taken as the solidifying point, or titer, of the fatty acids. The mercury will remain stationary at this point for several minutes, after which it drops again. Each titer test should be repeated and the difference between the two should not exceed .1° C.

The titer of a fat is the same as is designated as *hardness* in this country, the former expression being preferred in Europe. The determination is a very simple operation, yet the very slightest deviation from the established rules and the seemingly most unimportant external influences, such as a draft in the room, will influence the result to the extent of several degrees. It is also essential to be sure of complete saponification; a hundredth of a per cent. of fat left unsaponified in the operation sometimes means a degree of hardness more or less.

57. The method for making the titer test, given in *Quantitative Analysis*, while similar to the above, requires much more time to perform. In addition to this, the manner of stirring the fatty acids exercises a great influence on the result. The method just given enables the student to perform the complete operations for the titer in about three hours, and is that practiced both in packing houses and commercial laboratories, yielding quick and, at the same time, accurate results.

Table I is an empirical table compiled by Dalican, giving the percentages of stearic and oleic acids present in tallows yielding fatty acids of the solidifying points shown. The total yield of fatty acids is taken as 95 per cent.

58. Determination of Moisture in Tallow, Grease, Etc. — These determinations, together with the titer, or hardness, are the basis of commercial transactions in these articles. For the determinations, 20 grams of the average sample is weighed into a weighed porcelain dish. A small glass stirring rod is also weighed with the empty dish. The dish with the material and rod is placed on the water bath and heated, with occasional stirring, for 2 hours. It is then cooled in the desiccator and weighed. The loss in weight is the moisture contained in the sample. To insure accuracy, the material is again heated and occasionally stirred for half an hour longer, when it is again cooled and weighed. If no further loss in weight is observed, the grease or tallow is *dry* and all moisture has been driven off. The loss in

TABLE I

Titer, or Solidifying Point		Stearic Acid.	Oleic Acid.
C.	F.	Per Cent.	Per Cent.
35.0	95.0	25.20	69.80
35.5	95.9	26.40	68.60
36.0	96.8	27.30	67.70
36.5	97.7	28.75	66.25
37.0	98.6	29.80	65.20
37.5	99.5	30.60	64.40
38.0	100.4	31.25	63.75
38.5	101.3	32.15	62.85
39.0	102.2	33.14	61.55
39.5	103.1	34.30	60.80
40.0	104.0	35.15	59.85
40.5	104.9	36.10	58.90
41.0	105.8	38.00	57.00
41.5	106.7	38.95	56.05
42.0	107.6	39.90	55.10
42.5	108.5	42.75	52.27
43.0	109.4	43.70	51.30
43.5	110.3	44.65	50.35
44.0	111.2	47.50	47.50
44.5	112.1	49.40	45.60
45.0	113.0	51.30	43.70
45.5	113.9	52.25	42.75
46.0	114.8	53.20	41.80
46.5	115.7	55.10	39.90
47.0	116.6	57.95	37.05
47.5	117.5	58.90	36.10
48.0	118.4	61.75	33.25
48.5	119.3	66.50	28.50
49.0	120.2	71.25	23.75
49.5	121.1	72.20	22.80
50.0	122.0	75.05	19.95
50.5	122.9	77.10	17.90
51.0	123.8	79.50	15.50
51.5	124.7	81.90	13.10
52.0	125.6	84.00	11.00
52.5	126.5	88.30	6.70
53.0	127.4	92.10	2.90

weight multiplied by 100 and divided by the weight of the sample taken gives the percentage of moisture.

59. Determination of Impurities in Tallow, Grease, Etc.—The weighed material from the moisture determination is now dissolved in cold carbon bisulphide and filtered through a previously dried, weighed filter. The dish and rod are thoroughly rinsed by washings into the filter, removing from the dish and rod all particles of foreign matter, which must be weighed as impurities. The filter is washed free from grease by successive additions of carbon bisulphide until thoroughly free from greasy matter, when the filter, still in the funnel, is placed in the drying oven for about half an hour. The detached filter containing the impurities is then weighed. The increase in weight gives the amount of impurities in the 20 grams of material taken for test. This multiplied by 100 and divided by 20 gives the percentage in the material. The impurities are reported as “impurities insoluble in cold carbon bisulphide.” These usually consist of lime soap and extraneous dirt.

In the above determinations, it is always necessary to eliminate moisture before the impurities can be determined; if this is not done, the moisture in the material wets the filter paper, thereby preventing the carbon bisulphide from passing through.

60. Results of Tests on Products.—The results given in Table II were obtained in the packing-house laboratory on various products made, and on the cottonseed oils, for purposes of comparison. They may be regarded as regular, average results on the substances as produced in the packing house.

ANALYTICAL PROCESSES AND TESTS FOR FERTILIZERS

61. Introductory.—As previously mentioned, there are many tests and analyses made daily in the packing-house laboratories that, although not absolutely accurate, are sufficiently so to enable an immediate check, if necessary, to be

TABLE II

Product	Titer. Degrees C.	Iodine No.
Crude cottonseed oil (<i>for comparison</i>)...	35.05	110.42
Prime summer-yellow cottonseed oil....	36.65	106.52
Treated cottonseed oil (heated and blown somewhat).....	37.05	105.11
White (bleached) cottonseed oil.....	36.45	106.70
Cottonseed stearin.....		95.58
Lard compound (75 per cent. cottonseed oil, 25 per cent. oleo stearin).....	38.85	83.33
Lard compound (75 per cent. cottonseed oil, 15 per cent. oleo stearin, 10 per cent. tallow)	38.00	88.56
Lard compound (80 per cent. cottonseed oil, 20 per cent. oleo stearin).....	38.01	86.20
"Cotto" compound (cottonseed oil and tallow)	38.15	71.94
Lard (manipulated).....	39.35	67.77
Lard (manipulated).....	37.60	84.41
Prime steam lard (unbleached).....	38.65	64.85
Prime steam lard (old lard—pure).....	38.51	62.11
Prime steam lard (bleached).....	39.00	63.31
Prime refined lard.....	38.45	64.63
Pure leaf lard.....	40.50	57.39
Oleo stearin (bleached).. ..	50.65	23.68
Lard stearin (bleached).....	43.45	52.18
Tallow stearin (bleached).....	46.45	64.07
No. 1 tallow.....	42.95	
No. 2 tallow.....	42.48	
Butter-stock tallow (bleached).....	43.25	43.87
Mutton tallow.....	47.08	
No. 1 lard oil.....		64.07
Winter-strained lard oil.....		73.97
Prime steam lard (with 20 per cent. cottonseed oil).....	37.40	
Pure prime neatsfoot oil		70.53
No. 1 neatsfoot oil		67.41

placed on the various operations. There are many more or less crude and loose methods by which very rapid approximate results are obtained. It must be remembered, however, that these are for comparative and control results only, and are not intended for comparisons against accurate and commercial analyses made on the same materials, by outside chemists. When such results are desired, the analyses must be made by the most approved and accurate methods, and all loose or approximate working must be entirely eliminated from the operations. An example of this is when tankage or blood is sold on the basis of units of ammonia. While, for control results, it is immaterial, for example, whether the tankage contains 9.85 or 10.1 per cent. of ammonia, it is of the utmost importance, in selling, that the exact percentage of ammonia be known, as often the sale of perhaps many carloads may depend on it. Again, while for control purposes, all that it may be necessary to ascertain is whether or not a tallow possesses a titer of at least 42°, for sale purposes, where the price of the product is regulated by the exact hardness, the most accurate determination of the titer must be made as a check on the analysis made by the purchaser's chemist. It is always advisable, when time permits, to perform all operations in the various analyses with the greatest accuracy attainable, and to apply the rougher and cruder methods only when a quick, approximate result is demanded.

By the rapid methods that may hereafter be described, results are obtained that in no sense will stand against scientific and accurate analyses, yet they serve admirably the purpose for which they are intended. Most of the analyses made in the packing house are made with all possible accuracy, such, for example, as the determination of the Hübl iodine number, the free fatty acids of oils, tallows, etc. For the accurate determinations of the composition of fertilizers, the student should apply the methods given in *Quantitative Analysis*.

62. Scheme for Rapid Analysis of Fertilizer.—The mode of procedure in the rapid analysis of bones or tankage

in the packing-house laboratories for working results is carried out in the following manner:

The sample, if pressed tankage or wet bones, is dried in the usual manner to a degree that will allow of grinding through a mill. An ordinary coffee mill is well adapted to this purpose. If the percentage of moisture in the bones or the pressed tankage is desired, the sample is made completely dry and the amount of moisture evaporated calculated in the usual way from the loss in weight (see *Quantitative Analysis*). The sample is put through the mill until sufficiently fine to have an even, uniform, very finely divided mixture. Twenty grams of this mixture is weighed off in a weighed 3-inch porcelain dish and dried at 102° C. in the drying oven to a constant weight. This generally requires about 3 hours.

While this is drying, 1 gram of the sample is weighed off in a 4-ounce Erlenmeyer flask, or a Kjeldahl digesting flask,

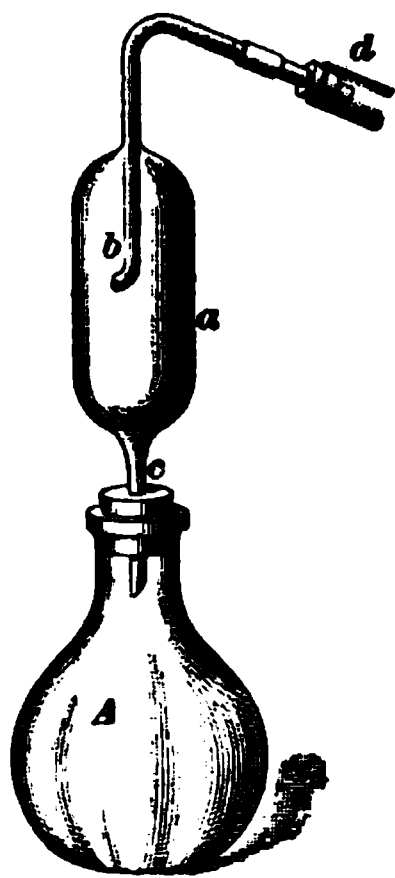


FIG. 9

and prepared in the usual way for the nitrogen determination by the Kjeldahl method. (For details of this method, see *Quantitative Analysis*.) For distilling off the ammonia, it is advisable to use a distilling bulb, as shown in Fig. 9. The liquid to be distilled is placed in the flask *A*. Into the stopper *c* is inserted the bulb. The vapors from *A* are freed in *a* from liquid and pass out through the curved tube *b* to the condenser *d*. The curved tube *b* prevents acid liquid from being carried over mechanically. During the drying and digesting of the sample, a portion (2.5 or 5 grams) is weighed off in a weighed porcelain dish, or

crucible, and the organic matter burned away. This residue is mainly calcium phosphate and is calculated as described later.

The sample for drying is weighed for loss of weight, which is noted as the moisture present. This portion, thoroughly dry, is now transferred to a filter-paper thimble, fitting

inside an extraction apparatus, and the fat extracted with light boiling naphtha of about 87° C. boiling point, or with rhigolene. The extraction is continued for about an hour, when all but traces of grease and fat are removed by the solvent. The operation is conducted as directed later. The digestion for the nitrogen determination has been completed during this time. This is now ready for the required determination of nitrogen by the usual method described in *Quantitative Analysis*.

63. There have now been determined moisture, nitrogen, phosphoric acid (or bone phosphate), and fat. By drying pressed tankage or wet bones over night, all these analyses may be made and reported within 5 or 6 hours. If the moisture of a dried tankage is 10 per cent. or less, then the material will keep in good condition when piled or sacked for shipment without heating or becoming lumpy. If the fat in tankage is less than 10 per cent., the work of cooking and pressing is shown to have been done well and economically. The amount of nitrogen and phosphoric acid in the tankage or other material determines its grade and, approximately, its market price.

For comparative purposes a tankage is always reduced to a moisture basis of 10 per cent. In this way it is immaterial whether a tankage is dried to 1 per cent. or 20 per cent. moisture for comparison with other tankages, when they also are reduced to 10-per-cent. moisture basis; the relative values may easily be seen. To obtain this result, the various percentages of the ingredients are multiplied by 90 and the result divided by the percentage of dry substance. For example, a dried blood analyzes 18 per cent. ammonia and 2 per cent. moisture. To obtain this on a basis of 10 per cent. moisture:

$$\begin{aligned} 18 \times 90 &= 1,620 \\ 1,620 \div 98 &= 16.53 \end{aligned}$$

Thus, the result may be expressed as blood of 10 per cent. moisture containing 16.5 per cent. ammonia, the equivalent of blood of 18 per cent. ammonia with 2 per cent. moisture.

64. Determination of Fat in Bones and Tankage.

The amount of grease left in the pressed tankage is the guide to the foreman or superintendent for correct, close, and profitable working in the fertilizer department. This analysis, though exceedingly simple, is one of the most important in the daily routine of the packing-house laboratory.

The method employed for this determination is as follows: The sample of bones, bone tankage, or tankage is dried in any convenient manner at a temperature not exceeding 102° C. The material is then reduced to a very fine condition and further dried to drive off all moisture. In the extraction of fats, it is very essential that the fertilizer material should be dry, or it will not be possible to extract the fat by reason of the moisture forming an impervious coating on the material, through which the solvent will not penetrate.

The dried fertilizer is transferred into a filter-paper thimble, made for extraction purposes, and the material pressed down with a wad of absorbent cotton, which serves to keep fine particles from being carried over into the flask containing the solvent. The thimble *c*, Fig. 10, containing the fertilizer, is inserted into the Soxhlet extraction tube *a*, which is connected with the usual condenser *d* and fitted into the weighed flask *b*, which is of about 100 cubic centimeters capacity. About 75 cubic centimeters of light boiling naphtha of 86° C. boiling point is used; or the extraction may be accomplished with rhigolene. The flask is usually heated by means of hot water or steam, as the danger from fire when heat-

FIG. 10
ing with direct flame is too great.

The heating is continued until all the grease or fat has been extracted from the fertilizer, which generally requires about an hour. If an exact determination of fat is desired, the heating may be continued for 2 or 3 hours, during which time the solvent will have been siphoned off about 100 times.

65. The apparatus is drained of solvent into the flask *b*. The thimble *c* is now removed, the flask again attached to the Soxhlet tube, and the solvent distilled into *a*. As fast as the tube *a* is filled with solvent it is detached and the distilled solvent decanted into a receptacle to be used again for further extractions.

The last traces remaining in the flask with the dissolved fat are driven off by placing the flask and fat in the drying oven and heating until the solvent is completely volatilized, which will not require more than an hour. The increase in weight of the weighed flask multiplied by 100 and divided by 20, the number of grams of material taken, will give the percentage of fat or grease extracted from the fertilizer material.

Ether cannot be used for extracting packing-house tank-ages, as this solvent will dissolve compounds, such as glyco-gen, etc., contained in the livers, which would give erroneous results. It has been found that light-boiling naphthas are the most satisfactory for extracting fat or grease from this class of material.

66. Rapid Method for the Approximate Determination of Bone Phosphate.—This method is largely practiced in packing-house laboratories on steamed bone, raw bone, bone tankage, etc. It is not applicable to fertilizers in general, as there are too many inorganic ingredients likely to be present. For packing-house products, being of organic origin, it is readily applicable.

The material is made in the usual manner into an average fine condition. Two and one-half grams of the substance is weighed into a weighed porcelain crucible of ample capacity, and the organic matter burned away. The heat of the ordinary Bunsen burner is sufficient for this. The material

should occasionally be stirred with a platinum wire, to expose the underlying parts to the air. When cooled in the desiccator, the ignited ash is weighed with the crucible and the weight of the crucible deducted. The ash is mainly tricalcium phosphate $Ca_3(PO_4)_2$, of a molecular weight of 307.9.

By multiplying the weight of the ash by $.86 \times 100$ and dividing by the weight of the sample taken, the percentage of bone phosphate (tricalcium phosphate) is obtained, the ash being considered as 86 per cent. bone phosphate. From constant comparative tests, this has been found to be very close to the actual amount present, sufficiently so for comparative purposes. The determination of *actual* phosphoric acid present in fertilizer, bones, etc. is carried out as described in *Quantitative Analysis*. For this analysis, $2\frac{1}{2}$ grams of tankage, or 1 gram of fine bone are the quantities taken.

COTTONSEED OIL AND PRODUCTS

(PART 1)

INTRODUCTORY

HISTORY AND DEVELOPMENT OF COTTONSEED- OIL MANUFACTURE

1. The histories of cotton, cottonseed, and cottonseed oil are entirely separate and distinct. The cotton plant and the use of the cotton fiber for weaving into cloth has been known for thousands of years, while the utilization of the seed for various economic purposes is of very recent date.

2. **Cotton Culture.**—The culture of cotton in the United States is said to have first been made at Jamestown, Virginia, in the year 1620. At the present time it is grown in all the Southern States from Virginia to Texas, and may be said to be the greatest industry of the South.

The seed is generally planted in March and the early part of April. It is thickly sown, in rows from 4 to 6 feet apart, by machine planters that distribute the seed economically and properly. The plants come up in about a week, and when an inch or two high are thinned out so that there are but one or two stalks in a hill, the hills being about $1\frac{1}{2}$ feet apart.

Careful cultivation is necessary to the proper development of the plant. The cotton plant, as is generally supposed, is not a hardy plant. It is very sensitive to climatic changes. Too much rain beats off the forming bolls and causes too rapid growth; after the bolls open, it causes "wet-weather blight," or rust. If the season is too dry, the vitality of the plant weakens and the bolls drop off and "dry rust" is caused. The plants grow and develop until killed by the frost. During this period; bolls form and open continuously, thus making the cotton-picking season a long one.

3. The cotton when picked from the open bolls is taken to the cotton gin, where, after more or less exposure to dry it, the lint is separated from the seed by means of saws that revolve at a high rate of speed. The seed is conveyed to the seed house while the cotton is removed from the saws by means of brushes and carried by a blast of air to the condenser, where it is compressed into bands and pressed into bales for shipment.

There are many varieties of cotton. Large quantities are grown in South America, Egypt, and India, and it follows that these countries produce large quantities of cottonseed.

As the cotton plant is grown primarily for the production of cotton, the value of the crop of seed is governed by the demands of the oil mills. In many localities, the demand exceeds the supply and the value of the seed is enhanced far beyond what was thought possible a few years ago.

4. Development of the Oil Industry.—The utilization of cottonseed in the manufacture of oil and other economic products has added millions of dollars to the value of the cotton crop, and the steps in the growth of the industry are of interest.

For a long time the Chinese have ground cottonseed for cattle feed, and incidentally recovered a little crude oil, which was used for illuminating and lubricating purposes. The first authentic record of the extraction of oil from cottonseed in the West was in 1783, when seed from the West

Indies was pressed in London with such satisfactory results that a prize was offered by the Society for the Promotion of Commerce and Arts to any planter in the British West Indies who should express a ton of oil from the seed and make 5 hundredweight of dry, hard cake fit for cattle food. This prize was finally withdrawn, as no attempt was made to win it.

In 1832, a small cottonseed-oil mill was operated on a small island off the Georgia coast, and 2 years later attempts were made to extract oil from the seed at Natchez, Mississippi. In 1847, further unsuccessful attempts to extract oil from cottonseed were made at New Orleans; one souvenir bottle of oil is said to have cost the proprietor \$12,000. In about 1855, the oil business was revived again at New Orleans, and also at Providence, Rhode Island. Since that date, owing to a study of foreign methods, the business has grown quite steadily. Previous to 1867, there were but four cottonseed-oil mills in the South and but seven in the whole country. The output of these mills was small and the products crude. At the present time there are from 600 to 700 mills in the South alone, each having a daily capacity of from 15 to 250 tons of cottonseed. About one-half of the total cottonseed production is crushed into oil.

5. The manufacture of cottonseed oil on a large scale may be said to date from about 1870. The crude and imperfect methods of that time have given way to the modern scientific and business methods. Previous to this time cottonseed was a positive nuisance, and the disposal of it was a serious problem. It had only a limited use as a fertilizer. Great quantities of it were piled on vacant fields, rendering the soil useless and barren for a time. Sometimes it was dumped into rivers, where it quickly rotted and gave rise to such stench that laws were enacted to prevent this practice. Much of the seed was disposed of by burning. At the present time the seed is eagerly sought for and has become of considerable source of revenue to the cotton grower.

Although the manufacture of cottonseed oil was carried on in England and Holland some time previous to its manufacture on a commercial scale in the United States, this country has outstripped the others both in the quantity and the quality of goods produced. The advantage that this country enjoys is in obtaining its raw material in a fresh state, while the foreign millers are obliged to use mostly Egyptian seed, which has suffered a long period of transportation. English cottonseed oil is made almost exclusively from Egyptian undecorticated seed, from which all the lint is removed in ginning.

6. Notwithstanding the fact that great quantities of cotton are grown in India, no cottonseed oil is made there. The Indian cottonseed is of a peculiar nature, smaller in size than American seed, and difficult to delint. Many failures have resulted in attempting to use it. Experiments are being made with delinting machinery, using emery wheels to grind off the lint, but their success is not yet demonstrated. There is also a heavy tax in India on manufactured oil, in order to keep the seed in channels of stock feeding and planting.

The shipment of American seed to England and Holland has met with rather poor success, owing to its bulk and great tendency to heat during transportation, which counteract other advantages.

7. Scope of the Cottonseed-Oil Industry.—Formerly the object of the oil miller was to recover the two principal products, oil and cake only, but through scientific chemical investigation it has become possible to recover many valuable by-products from what was once waste.

The tendency now is to concentrate under one management the handling of all products of the seed. It is common for cottonseed-oil refineries to work up their foots into various grades of soap, to recover stearin, to manufacture lard, butterine, and other edible products.

The cottonseed-oil business has been well advertised and none has been more systematically investigated and fostered

by the general government. The number of mills is rapidly increasing, but there is no likelihood of overproduction, as the demand for their products is on the increase, and the prejudice against the use of cottonseed oil as an edible compound has been largely overcome.

8. To thoroughly understand the manufacture of cottonseed oil and its various products, a thorough knowledge of all the different materials entering into the industry must be obtained and applied. Their bearing and effect on the various grades of products exercise a most potent influence; as, for example, the effect of caustic soda on crude oil, or the choice of a fullers' earth used to produce white oil.

Although the successful and economical operation of an oil mill depends to a large extent on the ability of the superintendent to produce a full yield of merchantable product without the assistance of chemistry, his efforts will be more reliable if coupled with scientific methods and refinement of working.

9. Application of Chemistry in the Cottonseed-Oil Industry.—The field of the chemist in the cottonseed-oil industry is broad and inviting. New possibilities for development are constantly coming up. In the mill, the application of chemistry in determining the grades of crude oil and the best uses to which it is adapted, and in indicating and correcting existing defects, whereby better yields and better products are obtained, attests its paramount importance.

The most important requisite in the production of cottonseed oil and its resultant cake at a profit is a modern, up-to-date equipment, under intelligent and scientific direction. From the time of receiving the seed, through the various operations of cleaning, decorticating, crushing, cooking, and pressing, careful and systematic working is necessary to produce high-grade, merchantable products.

In this Course these operations will be described in detail, and also the further treatment and manipulation by which the primary products are made into the various higher

grade articles, such as yellow cottonseed oil, white cottonseed oil, and articles of food into which cottonseed oil enters so largely, as butterine, lard compounds, etc. The most recent and approved method for the analysis of the products will also be given.

The chart, Fig. 1, shows to what extent the modern division of the cottonseed, as it comes from the gin, is capable. The industrial uses are but broadly indicated, the further details being given in the text.

COTTONSEED-OIL MILL

GENERAL REMARKS

10. The operations of the modern cottonseed-oil mill, as the machinery is now erected, are almost entirely automatic; the seed, starting at the sand-and-boll reel, is delivered by elevators and conveyers to the various other pieces of apparatus in turn, no manual handling being necessary until producing the oil and cake.

Various sized mills are in operation, from the small mill of 5 or 10 tons capacity per day of 24 hours, for plantation or gin use, to the mill with a capacity of 200 tons or more of seed per day. The mills most generally in use are those of one-press, 15 to 20 tons, commonly called a 20-ton mill; two-press, 30 to 40 tons; three-press, 50 to 60 tons. In each case they are called by the larger figure, up to several hundred tons capacity per day of 24 hours, the capacity being rated in tons of cottonseed that the mill handles. As stated above, the most usual and popular size at the present time is from one to three presses, there being more two-press mills built than any other size.

Like other expanding and flourishing industries, recent years have seen a tendency in the cottonseed-oil industry towards the erection of mills capable of handling several hundred tons of seed daily. While in many manufacturing

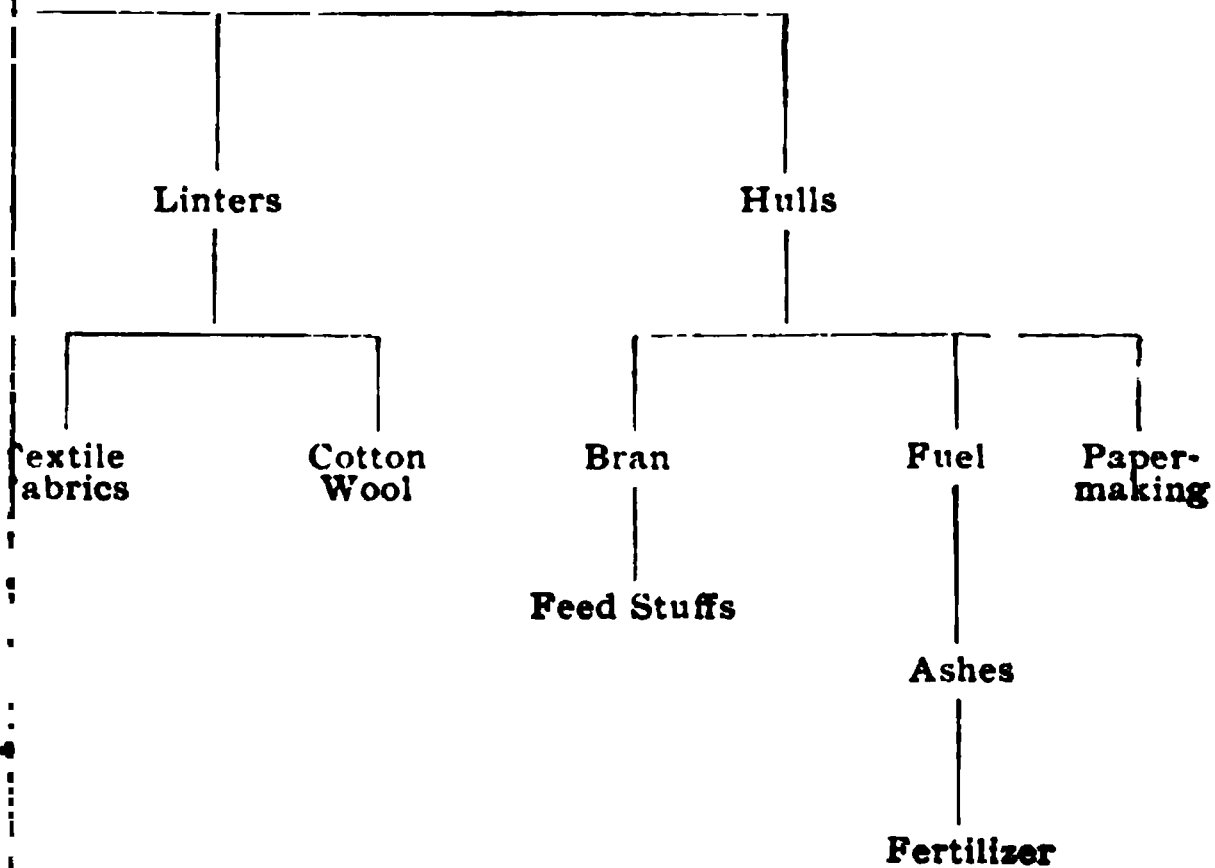
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industries such concentration is of great advantage, the wisdom of this proceeding in the cottonseed-oil industry is open to serious question. There is a limit to effective working in every manufacture, and with cottonseed-oil milling, this limit, in the opinion of many practical men, is in a mill handling about 50 tons of seed daily. Where the latter quantity of material is worked, every detail may be effectively looked after—a requisite for profitable working. Where hundreds of tons are daily worked in one establishment, the ever-varying condition of the raw material, the cottonseed, presents so many different phases, together with the many details of all the working operations, that the question naturally arises as to whether or not all the essential details can be given the constant vigilance and attention necessary for careful, profitable, and thorough working.

The usual number of hands employed in the oil mill is about twelve to fourteen in a one-press mill, from fifteen to twenty in a two-press mill, from twenty to twenty-five in a three-press mill, and twenty-five to thirty in a four-press mill. This proportion will not decrease much in the larger mills, as there is always more work in handling seed and the products in the large mills. The amount of labor required in a mill naturally varies with circumstances—the class of labor, the arrangement of the mill, and the capability of the superintendent.

For motive power in the larger mills, with the modern Corliss type of engine, it is customary to allow from 2 to 2½ horsepower to each ton capacity, and for small mills, with a slide-valve engine, from 2½ to 3 horsepower for each ton capacity. Additional boiler capacity must be allowed in all cases for cooking meats and for other uses.

11. General Principles of Manufacture of Cottonseed Oil.—The general principles that govern the manufacture of cottonseed oil may be briefly summarized as follows: Thorough screening and cleaning of the raw material, the seed, from foreign matters; thorough and good hulling or decortication of the seed; after this operation, as complete a

separation of the hulls and meats as possible; perfect and thorough crushing of the meats by the rolls; correct and careful manipulation of the crushed seed in the cooking kettles, with regard to temperature, moisture, etc.; sufficient and adequate pressure to the cooked meal for the extraction of the oil from the prepared material. The correct application of these principles embraces the whole operation of making from the raw cottonseed the crude cottonseed oil and cake.

The cost of working cottonseed in a well-equipped modern mill into its products—oil, cake, and meal—is from \$2 to \$3 per ton, the cost varying according to circumstances, equipment, and individual management.

12. Condition of the Seed.—Cottonseed as it comes from the gin is covered with a soft down, which adheres closely to the seed hulls. This is known as *linters*, in distinction from the long cotton fibers constituting the lint or cotton of commerce. The seed is of a size somewhat smaller than an ordinary bean and is encased in a tough, blackish husk, or hull. The condition in which the seed is received at the mill varies largely in the amount of contained moisture—the soil, season, and climate influence this to a marked degree—and it is from this cause that the methods in the most important part of the milling operations, the cooking of the meal, are subject to constant changes. Cottonseed from the first cotton picked or from unmaturing or frost-bitten plants gives a small yield of oil, of more or less poor quality. The best results in oil milling are obtained from mature seed.

STORAGE AND CLEANING OF THE SEED

13. Storing the Seed.—As the seed comes to the mill it is always mixed with more or less dirt, cotton bolls, sand, and other useless material, which must be separated before the seed is passed to the first process—that of delinting. The seed when unloaded from the wagons or cars is sent to the storage seed houses, or to the mill direct, through conveyers provided with perforated iron bottoms, through

which the accompanying dirt and sand fall to the ground or to another conveyer underneath, which carries this material to some desired place or receptacle.

The loss in weight from useless material in the seed depends very largely on local conditions in cleaning, the loss, if an average may be given, ranging from 3 to 6 per cent. This loss has reached in many instances as high as 10 per cent. In some instances the seed is unloaded from the cars and conveyed directly to the storage houses, where it is dumped automatically by the conveyers.

14. The seed storage houses hold the raw material, the seed, which is secured at the time of harvesting and ginning, for the operations of the milling season, the material being stored in bulk until wanted for making into the oil and cake. The bulk of seed in these storage houses frequently is from 30 to 40 feet high; but holding such a mass is very injudicious, as the great weight pressing on the under seed is very apt to generate heat and consequent fermentation.

Seed that has been trampled upon or partly crushed through handling is very prone to heat from oxidation of the exposed oil and increases the danger of deterioration in the bulk of seed with which it is mixed and stored. The more modern plan is to divide these storage houses into different compartments, each holding a moderate amount of seed, thereby lessening the liability of heating and deterioration by reducing the bulk that is kept in one compartment. It is very important to keep the stored seed cool and dry and to have the storage houses provided with good ventilation.

15. Sand-and-Boll Screens.—From the storage room the seed is carried by spiral conveyers, and by bucket elevators where necessary, to the **boll reel**, or, as it is commonly known, the **sand-and-boll screen**. This apparatus is shown in Fig. 2.

It is self-contained and varies in dimensions, suitable for the capacity required. The seed is fed into the reel, in which is a perforated cylinder revolving within the closed

box. The openings in part of the steel cylinder are larger than the seed, which falling through upon the shaker are

FIG. 2

evenly distributed over its surface. As they pass over the edge of it, they are caught by a current of air from the fan

near the screen and blown over a magnetic or precipitating field, which catches and retains any bits of iron, stone, and dust that remain in the seed. The screen removes from the seed, sand, dust, nails, and other foreign bodies, among the latter being cotton bolls—the capsules of which originally contained the seed cotton. These extraneous substances when separated pass from the screen through spouts into bags or boxes. This material is picked over by hand and an inferior variety of cotton obtained. This refuse material is technically termed **grabots**. Machines are at present being introduced for reginning this material, to obtain any cotton possible from it.

The machine shown in Fig. 2 is a very superior form of sand-and-boll screen, and through its working effects a great saving of wear and tear on the linter saws, owing to the almost complete removal of the metallic substances. The apparatus is also provided with an aspirator and dust flue, for the purpose of removing as much dust as possible. For two-thirds of its length the reel proper is covered with perforated metal or screen, of proper size to allow the sand and dirt to pass through; the remainder of its length has larger perforations for the seed to drop through. Provision is also made for carrying off the accumulated sand and dirt. The cleaned seed is carried by the current of air from the fan into conveyer boxes, by means of which the seed is automatically conveyed to the linting machines.

16. Size of Sand-and-Boll Screens.—Sand-and-boll screens of the different styles vary in size from 3 feet in diameter and 8 feet long to 5 feet in diameter and 18 feet long; where more cleaning capacity is desired than is afforded by these, it is customary to use one reel to remove the sand and another to remove the bolls and other foreign matter. The seed is usually first sent through the boll screen, or reel, that separates most of the matter larger than the seed; the latter then goes into the sand screen, in which the sand and dust that remain are quite thoroughly cleaned out. In some mills the sand-and-boll screens are reversed from the above order.

The wire cloth or perforated steel is adapted in size of mesh to suit the character of seed worked by the mill, as in some sections the seed is small with but a small proportion of dirt and trash, while in other sections of the country the seed is large and has much sand and dirt mixed with it. The latter is especially true in the Mississippi and Red River bottoms. Where the custom prevails of the ginner putting all motes and dirt from their condensers into the cottonseed, the loss in cleaning the seed is sometimes as much as 250 pounds to the ton. Recent regulations, however, have tended to greatly reduce this intentional mixing of worthless material with the seed. The loss in cleaning the seed and working it into its various products will vary from 100 to 250 pounds, depending on individual working systems.

17. Delinting Cottonseed.—Previous to the year 1887 there were practically only two methods of getting off the lint adhering to the hull of the seed, one being a chemical process that destroyed the fiber and the other a mechanical process that crushed the shell of the seed and extracted the kernel. By both of these methods the adhering lint was wasted, being completely destroyed by the former method and rendered useless by the latter, through being mixed with the broken shell.

The patented chemical process mentioned consisted in carbonizing the fiber on the hull by means of sulphuric acid, permitting its subsequent removal by washing easy of accomplishment. After repeated tests, however, it was abandoned, owing to the expense involved in the operation and the tendency of the sulphuric acid to carbonize and otherwise deteriorate the quality of the meats.

18. Cottonseed Linter.—The machine used to-day in nine-tenths of the mills in this country is shown by Fig. 3 and is known as the cottonseed linter. Through the use of this apparatus, the yield of oil from cottonseed has been very largely increased; at the same time the quality of the cake has been improved to a marked degree.

The linter is made with a capacity of from 3 to 20 tons of cottonseed every 24 hours, although claims are made for some newly patented linting machines for a much greater performance. These claims, however, yet remain to be satisfactorily demonstrated in producing a well-delinted seed.

19. This machine, shown in Fig. 3, removes most of the lint from the seed after it has been through the cotton gin,



FIG. 3

producing a merchantable quality of cotton, known as *linters*. The yield of this material depends wholly on the condition of the seed, wet or dry, the set of the saws, and the amount of seed admitted through the feed hopper. The cleaner the seed is made in the previous operations, the better will be the quality of the lint obtained and the more work the linter is able to perform without filing and gumming the saws. The modern linter is provided with an automatic feed *a*, which may be adjusted to any capacity, the quality of the lint, and the condition of the seed. Before the application of this mechanism an operator was constantly in attendance regulating the feed. When the seed was damp, it would go

through the linter very fast; when dry, it would be delinted very slowly, thus requiring constant observation by the operator. The automatic feed on a linter, together with a saw filer and gummer, materially increases the daily output of the oil mill.

20. Operation of Linter.—The working of the linter is as follows: The seed is fed into the linter at *j* and the supply regulated by an automatic feed actuated by the pulley *a*. The pulley *b* operates the cylinder with the saws. As the seed enters the linter, the supply is regulated for the saws by the mechanism operated by the pulley *g*. As the seed is fed to the saws, the latter, rapidly revolving, remove the lint from the seed. The lint is, in turn, removed from the saws by brushes on a revolving cylinder, actuated by the pulley *c*. This material goes through a passage to the slowly revolving gauze drum *d*, on which it collects and is wound upon the roll *e* to any convenient size.

When sufficiently large, the roll *e* containing the linted cotton is lifted bodily from the machine, withdrawn from the center of the cotton, and the roll again replaced on the linter. The seed, after being delinted by the saws, falls below and emerges at *h* ready for hulling. The lever *f* serves to start and stop the linter by tightening or causing to run loose the belt *i*.

21. The apparatus is provided with over a hundred saws arranged on an iron cylinder. The saws cut from the husk of the seed the lint, which then passes through a flue into the condenser, as has just been described. The "bat" here formed is collected into a roll by the swinging roll with which the condenser is provided. The seed passes from the linter with but a slight covering of lint, and is greatly improved for all purposes, as the huller will do better work on them in making a more complete separation of the meats and hulls; better oil and cake will be obtained than from seed not so treated.

Until the last few years all attempts to obtain in a commercially profitable way perfectly clean seed were unsuccessful. At the present day there are machines that remove

all the lint remaining after the above procedure. The short, woolly fiber removed is technically known as *delint* in the trade. The cleaner from lint the seeds are, the more perfect can be the separation of the hulls and kernels, or meats; consequently, a better yield of oil will be obtained from each ton of seed.

The quantity of lint cotton obtained from the seed, as in other things in this industry, varies largely; from 20 to 30 pounds are usually obtained from each ton of seed worked. A general average may be taken as 25 pounds.

22. Delint.—By reference to the chart of resolution of the cottonseed, Fig. 1, it will be seen that in addition to linters, delint also is obtained. This substance, as previously mentioned, is the very short, woolly fiber remaining on the cottonseed after the usual delinting process. The short fiber is obtained by practically polishing the seeds, thereby removing all lint except at the small depressed end of the seed. The seed obtained through this process resembles the Egyptian cottonseed.

23. Delinting Machine.—The machine for this purpose is of recent invention. It consists of four upright pillars that have midway between them a vertical shaft upon which four circular plates are fastened. These plates are provided with a more or less rough abrading surface. Three inches above these are circular brushes of the same size, each having a large circular opening in the center. The brushes are held stationary by means of arms attached to the uprights, while the shaft to which the plates are attached is revolving. A cylinder of sheet zinc encases the shaft plates and brushes, while a hopper between each pair of plates conveys the seed from one plate to the other through the circular openings in the brushes. The seed is fed in at the top of the machine and passes through the aperture in the center of the first stationary brush, and by the rapid revolution is rubbed between the brush and the first emery plate and part of the lint removed. It then passes outside the circumference of the first plate into another hopper, which sends it through

the opening of the second brush to the second emery plate, where the first process is repeated. In this way the operation is performed through the series of the four plates. The lint is removed by means of a fan, which carries it to the desired place. The cottonseed is thrown out at the bottom of the machine in a highly polished state entirely free from cotton, except for a minute quantity on the pointed end of the seed.

FIG. 4

24. This machine enhances to a considerable extent the value of the usual delinted seed, as the delint obtained is of considerable value for paper making, wadding, and even in some forms of fabrics. With the ordinary delinting process this lint is left attached to the hulls, which are always sold at a low price. For exportation purposes, seed of this character,

which is known as *bald* or *black seed*, is far more suitable than ordinary seed, as it occupies much less space and is far less subject to damage from heating and consequent deterioration, owing to the absence of lint.

Another machine for recovering the delint has been recently invented, but up to the present time it has been tried only on a limited scale. The seed treated by this machine is also almost entirely denuded of lint.

The quantity of delint obtained from a ton of seed has been shown by tests to be about 125 pounds, this in addition to about 25 pounds of the usual linters from the ginned seed; but further tests on this subject will enable those interested to judge more accurately of the capabilities and advantages of this process.

25. Saw Filing and Gumming Machines.—In close proximity to the linting machines are the necessary saw-filing machines and gummers. These are in constant use

FIG. 5

while the mill is in operation for sharpening and gumming the saws of the linters. They are, like most of the cottonseed machinery, practically automatic in action and are a necessary adjunct to every mill. Fig. 4 shows the filer and gummer engaging the saws of a linter. Fig. 5 is a view of the detached gummer, or machine for deepening the worn teeth of a saw. The sand and dirt on the seed that

escape elimination by the sand-and-boll reel rapidly dull the linter saws, rendering resharpening a frequent operation. In order to perform delinting properly, it is necessary that the linters be in perfect condition by having the saws kept sharp.

In working the linting machines, due care should be exercised not to overcrowd the machines, as in such cases the lint is very imperfectly removed with a consequent loss in the yield of linters. Another point dependent on this condition is that the more lint that remains on the hulls, the more of the finely crushed meats will be retained by these, with the inevitable loss of yield in oil and cake.

26. The **gumming** is done by a circular file *a*, Figs. 4 and 5, that rotates on an oscillating spindle and is so arranged that the file can be at work while the saw, which is being gummed, revolves. When the tooth on which the file is working has been sufficiently gummed, the file automatically withdraws and engages the next tooth. The process is continued until all the teeth in the saw are completed, when the machine stops automatically, and the gummer may be moved to the next saw.

HULLING THE SEED AND SEPARATING THE MEATS

27. Cottonseed Huller.—From the linters the seed goes by conveyers and elevators to the huller, which is conveniently situated on the second floor of the mill. This machine, shown in Figs. 6 and 7, has a capacity varying from 20 to 80 tons of cottonseed per 24 hours. Fig. 6 shows the front view of a huller and Fig. 7 the rear view of the same machine.

The hullers consist of cylindrical cases *a* containing horizontal knives, which vary in number from eight or nine to thirty, fastened upon their inner periphery. The cylinders are perfectly balanced, which is a prime essential to the good working of the machine.

The knives in the huller are made of the best steel and are tempered to the proper degree of hardness. Within the horizontal case *a* is a revolving drum upon which are fastened knives that, while revolving by means of the pulleys *b* at a high rate of speed, barely miss the knives upon the inner surface of the cylinder. The seed falling into the drum is rapidly cut to pieces, the mixture of hulls and seed falling to the separating apparatus below the huller.

FIG. 6

28. Cottonseed-Huller Feeders.—It is of great importance for good work that a regular and uniform feed of seed be maintained at the huller. Each knife should hull the same amount at each revolution of the cylinder. To meet these conditions, the **cottonseed-huller feeder** is used. This has a capacity for feeding any huller, and is so arranged that it can be connected to the hopper just above the huller. The feeder is capable, by use of a clutch, of shutting off the supply of seed at any desired time, in this way preventing, when the huller is about to be stopped, any

choking, thus leaving the huller free for starting up again. This apparatus feeds the seed uniformly and evenly over the surface in the huller.

The knives of the huller are of two kinds. A huller of 75 to 100 tons capacity is provided with seventeen cylinder knives 30 inches long and thirty concave knives. The latter are arranged so that they can be adjusted independently,



FIG. 7

and are held in position by setscrews. The smaller huller has similar knives, but less in number and about one-half as long. Hullers should be set often and the knives kept sharp, or the seed will not be cleanly cut.

29. Hulling, or more properly speaking, **decorticating**, is, and has always been, a very difficult process, owing to the varying conditions of the seed. When the latter is thoroughly dry and free from excess of lint, this operation is not so difficult of accomplishment, as the hull is then easily broken and the kernel, or meat, is loose and easily drops out

of the cracked hull. But when the seed is immature, damp, and soft, the hull will not be broken, but mashed so that it is hardly possible to get the meats separated from the hulls, unless with a great amount of labor. When the seed is in the latter conditions the knives must be very sharp and the seed fed at a slower rate, in order to get a satisfactory hulling.

Very recently a plant has been erected in Texas where the process of extracting the whole kernels from the cottonseed is in operation. This is claimed to be the only one of the kind in existence. The kernels are abstracted by the combination of machinery and chemicals recently invented. The capacity of this plant is claimed to be 100 tons of cottonseed daily. The husk-free kernels are mainly exported at the present time, but if the process proves to be all that is claimed for it, it means a great advance in the whole cottonseed industry. The clean kernels yield a high grade of oil and also a much greater proportion of oil than can be obtained from the usual decorticated seed. The residual cake, being free from the usual proportion of husk, contains an unusually high content of proteid matter.

30. Shaking Separator.—The decorticated seed from the huller when delivered to the shaking separator has a considerable amount of the whole meats extracted. This material is sent by means of a chute to the crushing rolls on the floor below. Using a shaker at the huller has been found to be a decided improvement over the practice of sending all the hulls and meats to the meat reel and separator for the entire separation there. By using the shaker, a large proportion of the quantity of meats is removed before the material goes to the separator. Much cleaner meats may thus be obtained, for unless the hulls are so separated, they will be more or less rolled together and intermixed in the revolving separator. In this way, the finely crushed particles of kernels will attach themselves to the lint on the hulls, thus materially reducing the yield of oil from the cottonseed material.

In some oil mills there are several hullers, and at each huller is a shaker about 8 feet long and $2\frac{1}{2}$ feet wide. By this device it is necessary to elevate to the separators only about half the material. Many mills use no shaker at the huller, but elevate the hulls and seed directly to the revolving separators by the usual conveyers; but, as previously mentioned, the use of the shaker at the huller is a great advantage in handling the decorticated material. The hulls with the remaining meats in them are then elevated to the meat reel and separator, which is also provided with a shaking separator, where the remaining meats are extracted from the hulls.



FIG. 8

31. Revolving Meat Separator.—This apparatus, Fig. 8, varies in size to suit capacity, the largest mills using those 5 feet in diameter and 18 feet long. The revolving screen is provided with such perforations as are suitable for

the class of seed being worked, a mesh of $\frac{1}{4}$ inch being most common. Either wire screening or perforated metal is used for the revolving screens. Perforations of $\frac{3}{16}$ inch are used in many screens, but as a rule better results are obtained with $\frac{1}{4}$ inch. Very good work is done by having a screen with $\frac{3}{16}$ -inch perforations in one end and $\frac{1}{4}$ -inch in the other. The meats and hulls are fed into the revolving reel at *a*, the former falling through the meshes into a spiral conveyer box *b* below the apparatus. The hulls, owing to their tendency to felt or to adhere together, are retained by the screen and are then delivered on to another separator, the finishing separator or shaker, where more of the meats are sifted out. These screens are arranged in a series, according to the capacity of the mill, the hulls being delivered from one to the other until they are free from meats.

32. Disposal of the Hulls.—The hulls when freed as completely as possible from the kernels are conveyed to the hull house, where they are pressed into bales for convenient handling, or are further conveyed, when not desired for sale purposes, to be used as fuel in the boilers. This use of hulls for the different purposes will be discussed later. In recent years the practice has prevailed of working more hulls into cottonseed meal, to increase the weight of the latter. This manipulation, or adulteration, if not prevented by legislation, will eventually assume such proportions that the whole cottonseed feed-stuffs trade will be irreparably injured.

CRUSHING THE MEATS

33. Crushing Rolls.—The separated meats from the previous operation are conveyed to the crushing rolls. Fig. 9 shows a set of five high-g geared crushing rolls. These rolls are also built three and four high. The rolls themselves, two of which are shown at *c* and *e*, are of chilled iron and vary in length, a common and usual size for a 75-ton mill being 48 inches long, and for a 100-ton mill, 60 inches. The bottom roll of the set is 16 inches in diameter;

the upper ones 14 inches. This arrangement of heavy rolls at the bottom is used in order to assist in driving the upper rolls by friction, which materially lessens the work on the belts and gearing. Each roll is driven by gearing or by belts from both sides; in the latter case they are provided

FIG. 9

with a belt tightener at each side. In a moderate sized mill the rolls are from three to four high. The roll shafts, excepting those of the bottom roll, rest in accurately fitted boxes, so that they are firmly held in position by the heavy iron frame containing them.

Three of the five rolls, *b*, *d*, and *f*, revolve in one direction, while the other two, *c* and *e*, which are between the first and third and the third and fifth, revolve in the opposite direction. The seed delivered into the hopper *a* at the top passes a small fluted or corrugated roll, which distributes the meats uniformly over the entire length of the upper roll, and by means of the guide plate is directed between the first and second rolls. The revolving cylinders, with the inward motion, rapidly draw in and crush the material, causing it to pass through or between to the opposite side. The crushed seed is again deflected by the slanting position of the cant board and made to pass between the second and third rolls, thus crushing it again in its passage. This operation is helped by the weight of the two upper rolls. In a like manner the seed is carried through the lower rolls.

34. When receiving its last crushing between the lower rolls, the seed receives the aggregate weight of the four upper rolls, which crushes it to a very thin, flaky fineness, thoroughly breaking the oil cells in the meats, a condition essential for obtaining a full yield of oil when the cooked meat is subjected to hydraulic pressure in a subsequent operation. The rolls are provided with balanced steel scraper knives, which prevent any crushed meats adhering to the rolls. The rolls are sometimes held together by heavy coil springs, which assist in crushing the oil cells of the meats and which will allow a hard, foreign substance, accidentally present, to pass through without injury to the rolls. The rolls revolve at various speeds, depending on individual judgment. From 120 to 180 revolutions per minute is the ordinary range, but as a rule the best work is done with the latter speed. A few mills run the different rolls at different speeds, claiming that by this method a better crushing effect is obtained, but the usual way is to have all the rolls revolving at the same speed.

35. It is very essential that all the rolls be true and in perfect alinement, or good work cannot be done. It is also equally important that all hulls be perfectly eliminated,

as in crushing any of the latter with the seed the meats will be but imperfectly disintegrated, and it will not be possible to obtain the full yield of oil from the seed. The mealy, crushed material should come from the roll in very thin flakes, with the oil cells of the seed thoroughly broken and mashed.

The condition of this material should be frequently examined and a close watch kept on the working condition of the rolls. By examining the crushed seed with a magnifying glass, the completeness of the crushing may be easily observed. It is the crushing operation that forms the basis for a good or poor yield of cottonseed oil, for if poor work is done at this point no subsequent operation can remedy it. The heavier the rolls and, within limits, the more there are, the better will be the crushing and, when the meats are cooked and pressed, the better will be the yield of oil.

COOKING THE MEATS

36. General Remarks.—The next operation, cooking, which brings the meats into such a condition that on pressing, the oil is readily liberated, is the most difficult and at the same time the most important of all the operations of cottonseed milling. There is no universal, or even usual, method of performing this operation, as each individual has his own way of handling the cooking kettles, or heaters, in order to produce, in his judgment, the best results. Such proceeding is necessary in this industry, owing to the fact that the physical characteristics of the seed are constantly changing. As the seed is fed to the kettles, one period may be passed with seed in a comparatively dry condition, when suddenly it will change to a moist condition. This may be the case when a fresh lot of seed has been crushed, or when one from a different section of the country follows the first lot. Also when a lot of seed that has been exposed to rains is crushed, difficult cooking is the result. Should the same treatment be given any of these qualities of meats as was

accorded the comparatively dry material, there is an inevitable loss in the earnings of the mills. The success attendant on cottonseed-oil milling depends in a great measure on the care that is exercised during the cooking.

The treatment of the crushed seed in the cooking kettle, or heater, is a matter of the greatest importance, and the strictest attention is always necessary, as this is the most critical point on which the profits of the mill depend. Carelessness or neglect here nullifies, to a great extent, the operations of rolling, etc., however efficiently these may have been performed. Proper temperatures must be maintained in cooking, together with having present the correct amount of moisture necessary for the crushed meats. As before mentioned, these conditions must be entirely governed by the nature of the seed under treatment, the heat and moisture being varied to suit existing conditions. The method of cooking the average seed will be described, together with the construction of the cooking heater, shown at *a*, Fig. 10, with the subheater *b*. This method, which has been adopted by many successful mills, has been found to produce excellent results, a yield of 43 gallons of oil per ton of seed frequently being obtained. The crushed meats from the rolls go, by means of the usual conveyers and elevators, to hoppers in connection with the heaters, the hoppers being so arranged as to hold just a proper charge of meal for the presses.

37. Cooking Heater. — This apparatus, shown at *a*, Fig. 10, is made in one solid casting and is steam jacketed on the sides and bottom. The sides are covered in many instances with insulating material, such as asbestos-faced hair felt, held in place by an outside lagging of sheet iron. The heaters are mounted on heavy iron frames, or legs, and are arranged with gear *c* to drive from underneath. Cooking heaters are also provided with overhead gear for driving, the arrangement being a matter of individual preference. Each heater is also provided with a charging hopper, which has a hinged and sliding gate for

filling the apparatus with material for cooking. The top of the heater is made in two sections, one of which is hinged and can be raised or slid to one side at will by the heater man, or cooker, to enable him to satisfy himself of the condition of the charge and also to give him access to the interior of the heater. The heater is also provided with a stirring apparatus, a heavy, forged-iron stirrer with arms that revolve for the purpose of keeping the meal agitated

FIG. 10

and not allowing it to stick to the sides or bottom of the heater. This agitation is most essential, for without it the meal would be burned in one part and undercooked in another. The heaters are furnished with mechanical appliances for breaking up the meal, which forms in balls, technically known as *water balls*. One arrangement is a set of knives that hang into the heater from the top; the balled meal, meeting these obstructions, is broken into pieces, so

that an even cooking may be obtained. In the heaters is a perforated steam pipe for putting moisture into dry meal.

Cooking heaters are made in two sizes, 52 inches in diameter and 72 inches in diameter. The former has a capacity of 20 tons of cottonseed per 24 hours and the latter a capacity of 40 tons of seed per 24 hours. The heaters are usually about 18 inches deep.

38. Operation of Cooking.—The method of cooking in the heater, after the meat is delivered from the hoppers, depends entirely on the judgment of the *heater man*, or *cooker*. A charge of about 700 pounds of crushed meats, or a quantity adapted to the size of the presses, is placed in the heater by pulling the lever of the hopper, covering the bottom to a depth of about 14 to 16 inches. It is not advisable to have too deep a layer of material, as this is much more difficult to cook evenly. The speed with which the stirring arms revolve in the heater depends on the number of them in the heater and the amount of material therein. A speed of from 45 to 60 revolutions per minute for a heater with three or four arms is a good average. With too slow a motion of the stirrers, the meal will get too soft and mush-like and will absorb too much moisture when steam is being injected into the material. The stirrers revolve close to the bottom of the heater, thoroughly mixing and stirring the meal as they revolve. The meal is constantly watched by the operator, who tests it from time to time, to ascertain its condition, admitting by means of the perforated steam pipe a sufficient amount of moisture to hold the material together when pressed.

The best results in cooking the crushed meats are obtained by cooking at about 80 pounds steam pressure. The time varies with different classes and character of seed. For comparatively dry seed a cooking of 14 minutes at this pressure gives excellent results. The time necessary for cooking dry meats varies from 12 to 20 minutes, and seed that is damp requires about 30 minutes without any addition of steam to it while cooking. Wet and frosted seed may require

a cooking in the heater of 40 to 50 minutes. Either too little or too much cooking gives a small yield of oil, so the importance of cooking exactly right is easily seen. An undercooked charge contains too much water and, in addition to this, the oil is not made sufficiently limpid and hot to flow freely from the material when placed in the press. When there is an excess of moisture present in the cooked meats, when placed in the press cloths, the latter are burst when subjected to pressure, causing much damage to this expensive material. If the material in the heater is overcooked, the oil, although of good yield, will be dark and of inferior character, a rank odor and taste frequently being imparted to it by the overheating. A test frequently applied by the operator is to withdraw a sample in his hand, pressing it together to ascertain whether it will hold together when tightly squeezed without oozing water, the material being sufficiently hot to be barely held without burning the hand. If the meats hold together, they are considered sufficiently cooked. If the material will not hold its shape, but falls to pieces, more moisture must be added to assist in forming a firm cake. When properly cooked, the meats are withdrawn either into the subheater *b*, Fig. 10, from which they are fed to the cake former, or into the conveyer that carries them to that machine.

39. The subheater is similar in shape and appearance to the cooking heater, the sides being covered with insulating material, as asbestos-lined wool felt. It is used for holding the cooked meats preparatory to making into cakes by the cake former. The material when sufficiently cooked may be dropped directly into the subheater and kept there for a considerable time without injury and at the proper temperature for pressing.

By the use of the subheater the whole charge from the cooking heater may be withdrawn at once, thus obviating all danger of having the last part overcooked when withdrawn directly for use in the cake former. There is, however, a wide divergence of views among oil millers regarding

the utility of the subheater, some claiming better results without its use; but in this, like in many other operations, individual experience is always consulted. For small mills, the subheater is practically a necessity.

FIG. 11

40. Many oil mills use the cooked material, without the intermediate holding in the subheater, employing the direct conveyer system shown in Fig. 11. This method eliminates

the charging heater, the cooked meal being dumped into the iron conveyer box *b* from the heaters *a* and carried direct to the charging hopper adjacent to the cake former *c*. The conveyer box is provided with screws, actuated by the pulleys *d*, which bring the meal to the charging hopper and cake former *c* under the conveyer, where it is pressed into cakes.

The pulleys *e*, with shafting, furnish the power to the gears *f*, the latter furnishing the motion to stirrers in the cooking heaters. The steel charging hoppers *g* are used to hold the rolled and crushed meats preparatory to being charged into the cookers or heaters. The set of four heaters shown in this figure can be extended to any capacity of mill by simply adding additional heaters and the necessary length of conveyer.

41. Water balls forming in the heater and subheater during the cooking are a great source of trouble and annoyance to the oil miller. When present in any considerable quantity, they cause the cottonseed cake containing them to have a spotted appearance. These balls of meal vary in size from $\frac{1}{4}$ or $\frac{1}{2}$ inch in diameter to 3 and 4 inches, and are made by the meal forming a nucleus and rolling, increasing in size, like a rolled snowball, until they can get no larger. The mechanical contrivances in the heater for breaking these balls do not always serve their purpose. These, however, are assisted by the conveyer in bringing the cooked meal to the cake former and also by the transferal of the material from the heater to the subheater, where the latter is used.

The water balls are practically raw meat on the inside, escaping cooking throughout, with the loose meal in the cooker. They are caused by excessive moisture in the meat. Many devices have been tried to eliminate them from the cooking of the meal, but up to the present time none have been entirely successful. Where these uncooked balls are pressed into the cake, the latter has a tendency to become quickly fermented and sour from the decomposition of the uncooked meat.

FORMING AND PRESSING THE CAKE

42. Cake Former.—The crushed meats when sufficiently cooked are withdrawn from the heater through the door or gate into either the subheater or the conveyer box, from either of which the material goes to be formed into cakes for pressing. For forming these cakes, the apparatus shown in Fig. 12, known as the **cake former**, is used. This machine, receiving the meal from the cooking heaters, forms it into cakes of the proper size to be inserted into the boxes of the presses. The rams *a* operating these machines

FIG. 12

are of various sizes, that in Fig. 12 being 18 inches, provided with an automatic cushioning device for the drop or return stroke. On the top of the ram is placed a meal box *b*, which is filled by a sliding meal carriage *c*, which registers with the discharge hopper of the heaters and is operated by a small steam cylinder, *d* in the rear, making its forward stroke at the will of the operator and its return stroke automatically. A 15-box oil press can be filled, by means of the automatic cake former, in less than 2 minutes. The machine works with clocklike regularity, and standing about waist

high, is operated by means of a lever e , which operates the machine by steam pressure; f is a counterbalance.

43. Forming the Cake.—The pan is placed on the former, into which it fits accurately; a press cloth of camel's hair about 6 feet long and sufficiently wide to accurately fit the press plates is placed over this, the center of the cloth being at the center of the tray. The meal is then charged into the tray and cloth, 3 or 4 inches deep, the ends of the press cloth quickly folded over, the pressure applied, when the cylinder ascends, raising the load and compressing it into the cake. After a few seconds the pressure is released, when the cake pressed in the cloth descends and the cake, with the cloth, is quickly inserted between the plates of the hydraulic press. The cakes as fast as made are placed, one after the other, after pressing in the cloths, into the different plates of the press. The whole operation occupies but a fraction of the time required to describe it. The cakes so molded by the automatic cake former are quite perfect in form, thus facilitating their adjustment in the oil press, and being even and uniformly pressed, give a greater yield of oil than was obtained in former years, when the old-style box was used. Hand cake formers are still in use in some small mills, but they are rapidly being superseded by the automatic formers. One machine of this description will furnish a sufficient supply of cakes for four to six presses as a minimum.

44. Oil Press.—This is also a very important piece of apparatus to the oil miller, the pressing being the stage where all previous operations show their efficiency or deficiency. There are several styles of hydraulic presses made, one of the most modern, a 16-inch steel press, being shown in Fig. 13. Hydraulic oil presses are usually made in two sizes, the 16-inch (meaning the size of the ram or piston c , Fig. 13) and the 12-inch. The former size contains from ten to fifteen steel plates a and the latter from seven to twelve plates, similar in design to those of the 16-inch, but

smaller in proportion. Two views of a steel press plate used in these presses are shown in Fig. 14.

The press, Fig. 13, is made of heavy castings to withstand the heavy pressure required, and is provided with raised edges *b* at the bottom for catching and retaining any oil that may drop down the sides while pressing. Each press is also provided with a hydraulic gauge for observing the applied pressure, a gauge pipe, and a change valve for its operation.

The press plates shown in Fig. 14 (*a*) and (*b*) have on the top (*a*) arrangements for the drainage of the oil. The lower side of a plate is shown in (*b*). In this plate there are but two parts, the main body *c*, made of one piece, and the "drainage plate" *d*, which is attached to the former piece by screws. The drainage may be of several



FIG. 13

kinds on the press plates, there being a great diversity of opinion as to the best form. The corrugated steel pan $\frac{1}{4}$ inch thick, perforated with $\frac{1}{16}$ -inch countersunk holes, is probably as good as any. Some oil millers use a drainage

with slots sawed out of a solid piece of steel or brass. In any event, the drainage plate must be kept perfectly free of loose meal or other extraneous matter, in order to give free passage to the oil from the pressed meal.

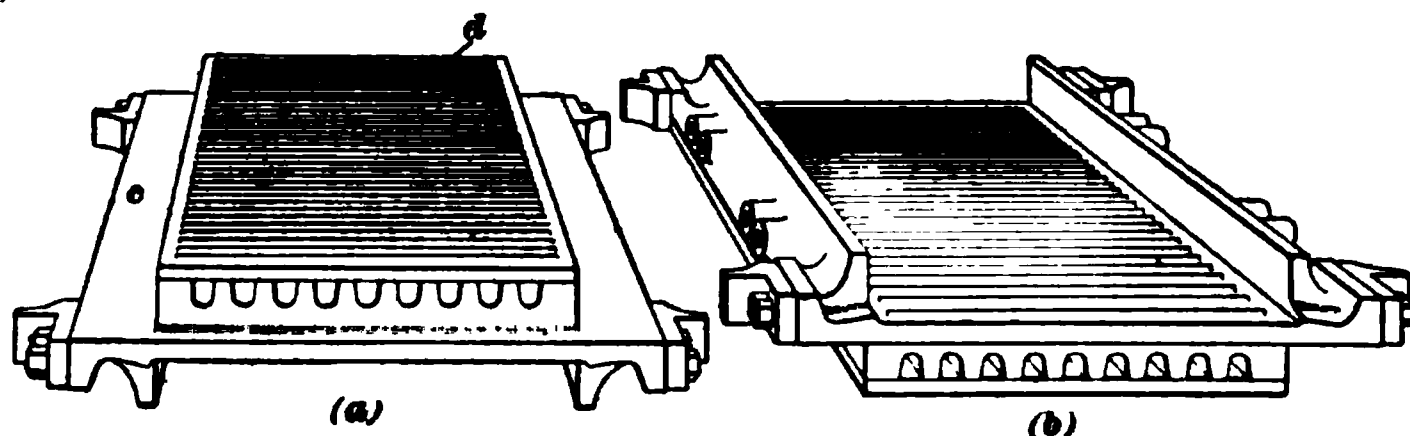


FIG. 14

45. The capacity of a press depends on the weight and number of cakes. The usual time which a press is run is 20 minutes, or 72 charges in 24 hours, but more than this is often accomplished. When the meal is well and properly cooked, the cakes made average from 12 to 14 pounds each, equal to about 20 tons of seed per day. It is also possible to run the presses on 15 minutes' time, or to make the cakes heavier. The latter course is not advisable, as better results are accomplished with a cake weighing about 14 pounds. The capacity of the hydraulic oil press depends also to a large extent on the capability of the operators and the percentage of oil designed to be left in the cake, as well as the loss sustained in the amount of press cloth damaged by excessive pressure applied.

46. Pressing the Cake.—The press being filled with the pressed cakes, the operator turns on the hydraulic pressure, and the ram rises and presses the material in the cloths with a pressure varying, with different operators, from 2,000 to 4,000 pounds per square inch. The lower cakes in the boxes are raised against the upper, and these against the heavy top plate of the press. As the mass becomes more and more compact, the oil begins to flow, at first slowly, and as the mass receives more pressure, the oil gushes forth in volumes from the material. It passes through pipes made

to receive it into large reservoirs beneath the press, from which receptacles it is pumped into settling tanks and in the modern mills through the filter press described later.

After the full pressure has been reached and most of the oil has been pressed out, the press is allowed to remain a few minutes to drain (from 7 to 10 minutes), when the pressure is withdrawn by reversing the control valve. The ram descends, carrying the lower parts of the press with it. The pressed cakes of meal, still hot from the cooking, are now withdrawn from the press and the cakes, stripped of the press cloth, are ready, after further treatment by drying, to be packed as cakes or ground into meal.

47. The crude oil, where there are no filter presses used for clearing it, is pumped to storage tanks and allowed to settle for some days. Ordinarily, from 4 to 6 days are required to effect a good settlement of the oil, but this, like many other operations in the oil-milling industry, is a matter largely governed by circumstances. The further treatment of the crude cottonseed oil for industrial purposes will be described in subsequent articles.

FIG. 15

48. Hydraulic Pump.—The pressure is applied, as before stated, by hydraulic pumps especially made for this

purpose. These have been found far more serviceable than power pumps, etc. The pump is always provided with an automatic regulator and safety valve. The hydraulic-pressure regulator serves to admit only enough steam to move the pump at an even speed; as the pressure rises, it automatically admits more steam until the required pressure is reached, when it automatically closes, and the steam being entirely cut off, the pump stops. The pump thus requires but very little attention. Fig. 15 illustrates a modern hydraulic pump with the automatic pressure-regulating valve *a* shown to the right. One of the steam cylinders is shown at *b* and a water cylinder at *c*.

49. Most mills are equipped with a pressure accumulator, which acts as a reservoir for the pressure generated by the pump. The pressure accumulator is used for both low and high pressure. The low pressure is used on the press to take up the slack of the ram before the heavy pressure is applied. By then simply turning a valve, the maximum pressure of the accumulator is applied to the press, thus rendering its working very rapid and effecting a great saving of time; there is also a great saving in the matter of press cloth.

The pressure necessary to extract the oil has been the subject of much discussion. The best work, however, when the previous operations have been properly carried out, is obtained by applying a pressure of 3,500 to 4,000 pounds per square inch. Some crushers apply but 2,500 pounds, but this pressure, while extracting a great portion of the oil from the crushed seed, leaves too great a percentage in the pressed cakes.

50. Camel's-Hair Press Cloth.—This is a woven fabric made from camel's hair. With the use of hydraulic presses, with the accompanying enormous pressure to which the press-cloth material is subjected, camel's-hair cloth has been the only one found suitable for good and economical working in the extraction of oil from cottonseed. The fine, long

camel's hair, tenacious and elastic, is woven into cloth, resembling somewhat in appearance ordinary, but very closely woven, bagging, which is capable of withstanding a pressure of from 3,500 to 4,000 pounds per square inch without damage.

A press cloth of this material, if used with intelligence, and properly prepared meal only is pressed, will last a whole season. But when meal containing an excessive amount of moisture is placed in these cloths, they are more than likely to be burst and ruptured at the moment the heavy pressure is applied. With improper cooking and pressing of cottonseed meal, no matter how perfect the press-cloth material may be, its usefulness will very rapidly be destroyed. Many times a new press cloth has been rendered useless for further pressing during the first few charges of the press, owing to these conditions. A press cloth made from good, long staple camel's hair will last, when properly used, at least 6 weeks, the mill running the usual 24 hours, before it is rendered unserviceable.

51. The item of expense for press cloth is always heavy in cottonseed milling, as the initial cost of the cloth itself is considerable. It is difficult to give any average cost, per ton of seed milled, for this material, owing to the fact that it is subject to so much difference in individual working. While one mill may crush for an entire season at a cost of 6 cents per ton of seed, another mill in the same locality may have the cost per ton for press cloth as high as 25 cents. Where such an excessive cost as the latter obtains, in most cases it is indicative of careless working. Where the cost per ton of seed crushed exceeds this figure, there is something radically wrong with the operations of the mill, most probably with the cooking or with the pressing. A reasonable figure for press cloth under ordinary conditions should not exceed 12 to 15 cents per ton of seed worked. Many successful mills work the whole season with an average cost for press cloth of about 10 cents per ton of cottonseed crushed.

The worst damage occurs to press cloth when poorly crushed seed, very hot material, and watery cooked meal are pressed. Any or all of these features exercise a very great influence on the life of the camel's-hair cloth.

TREATMENT OF COTTONSEED CAKE

52. Preliminary Treatment.—The cakes when taken from the press and stripped are very hard and solid, like a piece of board. They are ordinarily 14 by 32 inches, the regulation size of cottonseed cakes. They are piled on trucks and sent to the cooling room, where they are cooled as rapidly as possible, piling on racks being resorted to, to effect this. A further purpose of this procedure is to give the material time to dry out any superfluous moisture, a necessary precaution to prevent the cakes from souring and molding. After remaining until cool, which requires from 12 to 24 hours, the cakes are fed into a machine known as the *cake breaker*.

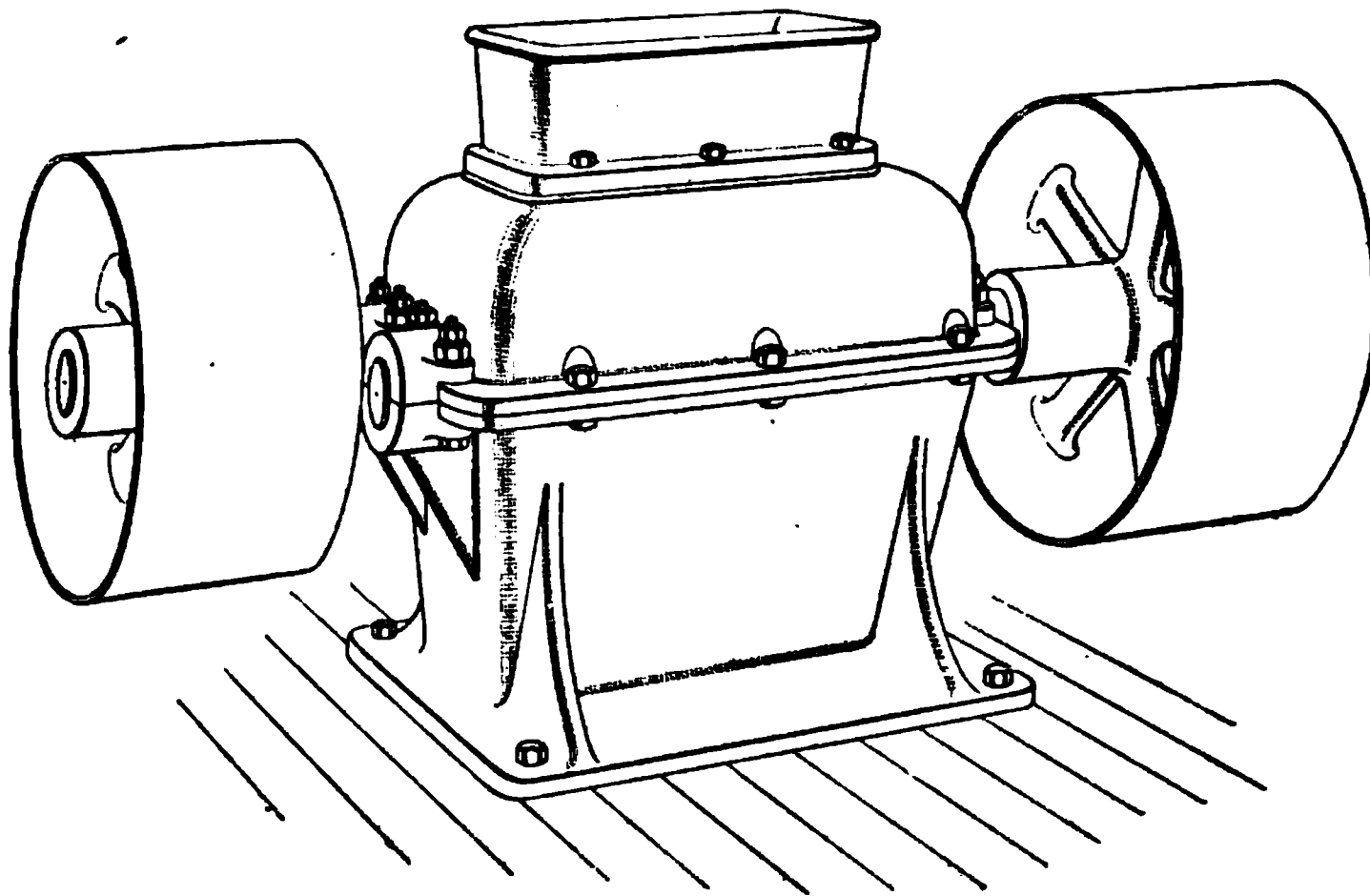


FIG. 16

53. Breaking the Cake.—The cake breaker, Fig. 16, revolves at a speed of 300 to 350 revolutions per minute. The cakes are fed into the cake breaker as fast as the machine

will receive them and broken into pieces about the size of a nut, so that they may be conveyed mechanically to the mill for grinding into meal.

There are many forms of cake breakers and many machines have been constructed for this purpose that have failed to answer the rigid requirements of this piece of apparatus. The cake breaker must be very strongly constructed and made so that soft and broken cake will not choke it. The cake breaker can be run with one belt where there is sufficient power.

54. Grinding the Cake Into Meal.—From the breaker, the broken cakes are conveyed to the grinding mills, which are in many cases burrstone mills, but more commonly and much better are attrition mills, which grind the cake into the cottonseed meal, which is then sacked for sale purposes or used for fertilizer, etc.

In some cases, the cakes are trimmed free of ragged and uneven edges and packed in bags for export, some foreign trade preferring the material in this condition, doing their own grinding when desired.

COTTONSEED OIL AND PRODUCTS

(PART 2)

REFINING CRUDE COTTONSEED OIL

INTRODUCTORY

1. As previously noted, the condition of the seed influences to a great extent the quality of the crude oil produced. Where cottonseed has been stored for any length of time, and thereby becomes heated, owing to a damp condition, the free fatty acids resulting from decomposition will appear in the resulting oil to a marked degree. The higher the oil is in free fatty acids, the less will be its market value, the greater the shrinkage in refining, and the lower the grade of the finished product.

Crude cottonseed oil as obtained from the oil press is a thick fluid varying in color from yellowish brown to a dark ruby or blackish red, according to the nature and condition of the seed from which it has been pressed. It contains much moisture, or water, and impurities consisting of fine meal, "mealy matter," coloring matter termed *Gossypium*, mucilage, and more or less tarry matter. Most of these are due to the application of heat in pressing the oil from the cooked seed. If it were commercially profitable to press

cottonseed by the cold process, an almost white oil could be obtained, in common with most vegetable seed oils. Cold pressing is carried on to a limited extent in France, but the price obtained for the product is commensurate with its advanced cost of manufacture. The yield by the cold-pressure system of extracting cottonseed oil is far below that from most other vegetable seeds, such as linseed and rape.

The yield of cottonseed oil by the usual cooking ranges from 12 per cent. in poor working and from poor seed, to 17 per cent.—the latter in exceptional cases and from good seed. The general average yield may be taken as 15 per cent., or 300 pounds of oil to the ton. The general trade requirements of crude oil are explicitly set forth later.

While the oil miller may not readily be able to dispose of his crude product, as it does not fill the usual trade requirements, he is enabled by means of the oil refinery to convert it into oils of higher grade, and, if provided with the necessary equipment, into finished articles of which cottonseed oil is the base.

2. Crude oils containing less than 1 per cent. of free fatty acids are used in the manufacture of butter oils. Only selected seed is used and great care is taken in the process of manufacture in order to avoid any unpleasant odor or taste.

Crude oils testing between 1 and 2 per cent. of free fatty acids are refined in the regular way to a prime yellow oil and should grade as such.

Crude oils containing more than 2 per cent. of free fatty acids are unsuitable for edible oils, as their original reddish-yellow color does not disappear by refining.

Crude oil may have as high as 7 or 8 per cent. of free fatty acids in exceptional cases. Such crude oil should be handled to produce as high a grade as possible with a minimum amount of loss in refining. Oil of this nature is suitable, naturally, only for industrial and not for food purposes.

The loss in the refining of crude cottonseed oils varies according to many circumstances. The greater the amount

of free fatty acids, the greater will be the loss in refining. The average loss in the refining of good cottonseed oils approximates from 8 to 10 per cent. In many cases this loss is much less, and likewise in many other cases much greater.

APPARATUS AND METHODS USED IN REFINING CRUDE COTTONSEED OIL

APPARATUS FOR REFINING CRUDE COTTONSEED OIL

3. The methods used in this country for the clarification or refining of cottonseed oil vary considerably in the details, but all are based on the use of a caustic alkali, employed either alone or with certain adjuncts. The alkali almost universally used is caustic soda, and the adjuncts used in connection with refining are permanganate or bichromate of potash, and sulphuric or hydrochloric acid, chlorine gas, or oxygen. The use of caustic soda in solution, however, is now almost exclusively the only reagent used in the largest refineries, but at times this is supplemented by the other agencies.

The apparatus necessary for refining, shown in Fig. 1, consists of a lye tank *A* for holding the solution of caustic soda, a refining tank *C*, a soap-stock tank *D*, and a finishing, or rectifying, tank *E*. The lye tank is placed some distance above the refining tank. The former is made of boiler iron and is thoroughly impervious to leakage. The tank is fitted with pipes $1\frac{1}{2}$ inches in diameter, controlled by a cock for regulating the supply of caustic soda. A perforated sheet of iron, or a sieve, *b* is placed under these pipes and over the refining kettle, covering about three-quarters of its surface. This enables the lye to be evenly and finely distributed over the surface of the crude oil and insures a thorough, intimate mixing of oil and lye by agitation, which is a most essential part of the operation. The lye is in many cases added to the crude oil by means of a perforated pipe extending across the refining tank.

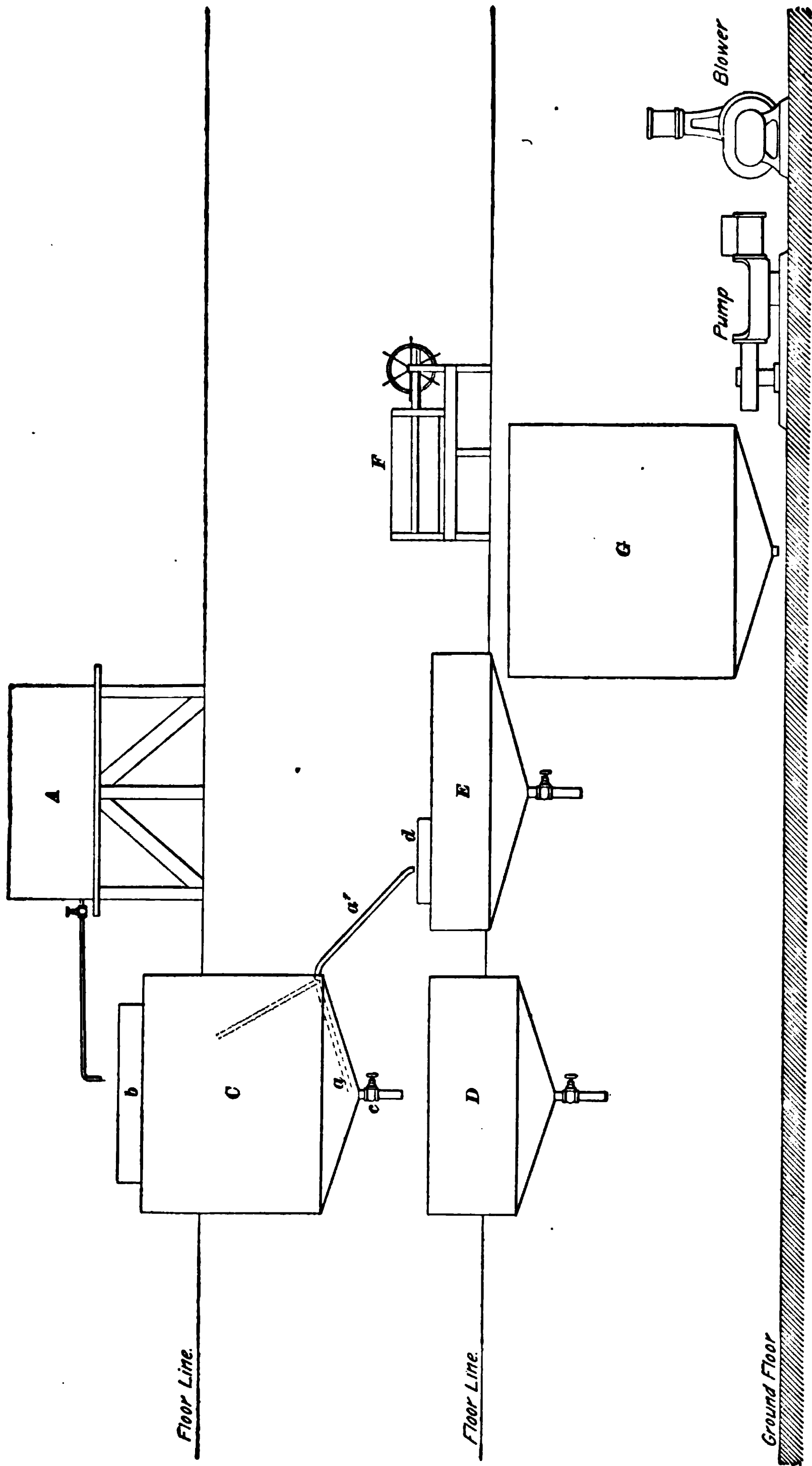


FIG. 1

4. Refining Tank and Its Accessories.—The refining tank is circular in shape, of sheet iron, and of size adapted to the requirements of the plant. They are usually made to hold from 7,000 to 12,000 gallons. The diameter is about one-third greater than the depth. There are several methods of agitating the oil while it is under treatment; the principal ones consist of agitation by the *air blower*, by means of the *paddle wheel*, and by the *screw propeller*.

5. The air pump, or blower, is very largely used for this purpose, but it has been discarded by some refiners using the caustic soda alone, for the reason that the heavier caustic solution resists much more than the lighter oil the agitation from a stream of air forced into the mixture by the blower. This having been fully demonstrated, it is obvious that there is imperfect agitation and mixing, and a consequent loss in greater shrinkage by the use of this method.

6. The paddle wheel, similar in style and shape to the ordinary side wheel of a steamer, is made of a size to practically fit the refining tank. It is actuated by a pulley on an extension of its shaft extending through one of the sides of the tank. When the wheel is set in motion, it stirs the oil and caustic solution and effects a thorough mixture.

7. The screw propeller is like the ordinary type of ship propellers, with a pitch designed to throw up the oil at an angle a little less than 45° . It is placed at the bottom of the tank and is driven from above, its shaft descending into the tank. The propeller need not be over one-third the diameter of the tank, as when in action it creates a motion that effectually agitates the oil and mixes it intimately with the caustic solution.

8. The refining tank is provided with closed steam coils $1\frac{1}{2}$ inches in diameter, placed at the bottom of the tank and continuing up along the sides to within two-thirds of the top. These pipes serve to supply the necessary heat to the oil when ready to work. In addition to these, open steam

pipes are also placed in the bottom of the tanks, as live steam is a helpful adjunct in refining. The bottom of the tank is made slightly in the shape of an inverted cone, to facilitate the discharge of the stock by means of a large cock placed in the center, or apex, of the bottom. The refining tank is placed just above the soap-stock tank, so as to discharge the formed foots by gravity.

9. When air is the mode of agitation used, the same pipes used to inject air may also be made available to inject live steam by simply making the necessary connections outside the tank. When it is desired to pursue such a course, the following disposition of the pipes should be made: the pipe forcing the air from the compressor into the oil should enter, with as few sharp angles as possible, the center of the tank at the top and descend vertically therein to the bottom. The main pipe should be at least 4 inches in diameter, 6 inches being still better for this purpose. From the end of this pipe a number of 1-inch pipes emanate, extending along the bottom of the tank, the pipes being in length about one-third the diameter of the tank. They are perforated with holes $\frac{1}{8}$ inch in diameter, near the under side and at an angle of 45° , so as to give a scouring force to the jets of air. The area of the openings in the small pipes should not exceed that of the main supply pipe or the blower will not be capable of effective work. This is a most essential point. The ends of the pipes are capped, with a perforation in each cap. The appearance of the arrangement of pipes is similar to that of a spoked wheel, the tire being absent.

The mechanism arranged in the above manner creates an upheaval of oil in the center, while the oil at the sides is drawn in near the bottom. The entire mass, in other words, flows up in the center and down at the sides, the operation thus causing a rotary motion from the center to the circumference. In this way the maximum utility from the air is obtained.

10. Soap-Stock Tank.—This tank is placed directly beneath the refining tank. It is circular in shape, made of

sheet iron, and is provided with a closed steam coil. It has usually the same diameter as the refining tank, but is only half its depth. The bottom is also made similar to that of the refining tank and has closed steam pipes, the purpose of which will be explained later.

11. Finishing, or Rectifying, Tank.—This is a large, flat tank provided with closed steam coils $1\frac{1}{2}$ inches in diameter and with perforated air pipes. Its depth should not exceed 3 feet and that of the oil placed therein $2\frac{1}{2}$ feet. The tank is usually made of sufficient capacity to contain the oil from one treatment of crude oil. The general arrangement of the apparatus is shown in Fig. 1.

The above apparatus, with the air blower, pump, and necessary power, comprise that necessary for the refining of crude cottonseed oil. The general operation is carried out in the following manner, more specific details being discussed in later pages.

12. Refining Crude Oil by the Caustic-Soda Method. The general arrangement of the refinery is shown in Fig. 1. The crude oil is heated in the refining tank to about 80° F., sufficient room being left therein for the solution of alkali and for gentle agitation. The agitator is set in motion, and when fairly started is regulated so as to give the oil a slow and regular rolling motion. The cock on the caustic-soda tank shown at *A*, Fig. 1, is then opened and the lye allowed to drop slowly and gradually through the screen *b* or through a perforated pipe into the oil in the tank *C*. The quantity of lye necessary depends wholly on the quality of the crude oil, from 5 to 15 per cent. of the volume of the oil generally being used; this is also governed by the strength of the lye used for refining purposes.

During the mixing of the lye with the oil, the operation is closely watched to ascertain when sufficient alkali has been added. The appearance of the oil changes in a few minutes after the introduction of the soda solution, the bright yellow froth, always present on the oil, gradually disappears and that remaining has a tendency to bleach.

The oil assumes a brownish hue, becomes more fluid, and gradually turns blackish in color. This change will take place about 15 or 20 minutes from the starting of the operation. A sample now withdrawn in a ladle will show a clear, bright oil intermixed with small blackish clots. This is the critical moment; the experienced operator always relies on his eye to judge the "turning" or "breaking" in the oil. The ordinary and usual method is to dip with the hand a portion of the oil, when the separation in the palm is easily observed. When this condition is observed, the flow of the caustic-soda solution is immediately shut off and the agitation reduced to maintain a long, rolling motion. The oil is now heated; at a temperature between 100° F. and 120° F. the oil will break well and the agitation will cause the agglomeration of the particles of soap stock, so that the oil will settle clear. The time required for the refining operation is about 30 to 45 minutes.

When the oil is seen to be settling well, the agitation is wholly stopped and the refined oil allowed to settle. It is the general practice to allow the oil to settle over night, although some refineries withdraw the supernatant oil after 4 or 5 hours' settling. In treating prime crude oil, the temperature in the refining tank must not exceed 130° F.

13. The clear oil is now drawn off from the tank by means of the siphon *a*, through the pipe *a'*, into the finishing tank *E*. The screen *d*, covered with gauze or cheese cloth, serves to catch and retain any fine particles of soap stock that may be carried over when the siphon is withdrawing from the refining tank the oil just above the settled soap stock.

In the finishing tank the oil is heated to 150° F. and blown with air until a sample, withdrawn in the tester or sample bottle, remains clear and unclouded when chilled to 60° F. When this condition is observed, the oil is free from moisture and is then allowed to settle or is pumped through the filter press *F* into the storage tank *G*. It may be barreled when cool as summer yellow cottonseed oil.

14. While the foregoing is the general plan followed, many mills work out many of the details in a different manner after the crude oil is settled. For example, in some refineries, after the oil is sufficiently refined, it is treated with a weak brine, made by dissolving 1 ^{part}~~per-cent.~~ of salt in ^{99 parts} hot water. This is agitated with the freshly refined oil, which procedure materially assists in the clarification. Some refiners after withdrawing the yellow oil subject it to a thorough washing with fresh water to remove all possible traces of alkali. This course is followed particularly with oils desired for edible uses.

By means of the filter press, the operation of settling or removing the impurities may be materially shortened and other advantages gained, which will be described later.

15. Refining Crude Oil by the Caustic-Soda, Bichromate-of-Potash, and Sulphuric-Acid Process.—As has been stated, the caustic-soda process is sometimes used with other adjuncts in the process of refining crude oils. With oil extracted from heated seed, or seed otherwise unsound, the use of caustic soda alone is not capable of producing a bright yellow oil from the crude article, and hence the necessity sometimes arises for supplementing the effects of caustic soda by that of other and more potent agencies. The method most generally practiced is the following:

The crude oil is given the preliminary treatment with the caustic-soda solution, but as soon as it is thoroughly cleaned and is of an off-yellow, or reddish, cast, it is transferred into another clarifier and there thoroughly washed with free superheated steam. This serves the double purpose of both cleaning and bleaching the oil. It is then allowed to settle, after which it is drawn into a lead-lined tank for subsequent treatment. The lead-lined tank is provided with copper steam coils and agitation is effected by means of the air pump and perforated lead pipes, arranged as in the ordinary refining tank. The oil having been placed in this tank is slowly heated to 100° F.

A 10-per-cent. solution of bichromate of potash having been prepared with filtered water, a quantity of this, equal in volume to from $2\frac{1}{2}$ to 5 per cent. of the oil, is sprinkled over the surface of the oil while it is being agitated. As soon as the oil appears of a dark green or even greenish tinge, the supply of bichromate solution is stopped. The agitation is continued and chemically pure sulphuric acid, diluted one-half or three-quarters with water, is now slowly added to the oil. The condition of the oil is tested from time to time by means of the test glass. When the precipitation is complete, as may be easily seen in the glass, the supply of acid is stopped, the air agitation shut off, and the mixture allowed to rest for a few hours.

16. The underlying acid water is withdrawn from the bottom and the oil washed with warm water until all traces of the acid have disappeared, when the material is sent to the rectifying tank, as in the ordinary caustic-soda process. Sometimes previous to washing the oil with water, a washing with weak lime water is resorted to, but poor as the oil may be, this operation is injurious to the flavor of the finished product. Consequently, it is used only in the case of very poor oils. The best method ordinarily is to use only warm water in a tank with paddles, which give the oil a thorough beating and washing. This necessitates the decanting of the oil into one of the iron clarifying tanks. Air agitation may be used for this, but the other method is considered more reliable and effective. If it is the intention to make from such crude oil a white oil, the refining in the first instance with the caustic soda is pushed to the straw color of prime refined cottonseed oil. The oil should then be steamed and finally treated as just described. This obviates the necessity of using an excessive amount of acid.

17. Refining Crude Oil by the Permanganate-of-Potash Method.—In this process, caustic soda, permanganate of potash, and sulphuric or hydrochloric acid are used. Permanganate of potash with some oils gives better results

than bichromate, and enables the refiner to make a palatable oil out of an inferior article, otherwise suitable for crude industrial purposes only. The same may be said of sulphuric and hydrochloric acids used in connection with the permanganate. With certain oils, one does better work than the other, and nothing short of an actual laboratory test can guide the operator in this matter. Such tests should be made on all oils of this class before attempting to work with the usual large quantities.

The method of treating the oil by the permanganate process is carried out in the same manner as has been described. The proportions of the ingredients used are also the same as in the bichromate method.

The bleaching of cottonseed oil by means of chlorine gas or oxygen is not done in many of the refineries of this country, the caustic-soda method being employed by far the greater number, as a preliminary to the bleaching of a yellow oil.

18. Refining or Bleaching Crude Oil by Ocher. Among the various methods of bleaching cottonseed oil is a patented process in which the use of an iron ore, such as limonite or hematite or ocher, is used. With some oils it does excellent work, but the patent covering it probably acts detrimentally towards its more general adoption. This method besides being applicable to cottonseed oil is also used with great success for the refining of linseed, rape seed, and analogous vegetable oils.

The process is carried out by the use of the above-mentioned substances or of any similar substance of the nature of a pigment that does not injure the oil as an article of food. By mixing and agitating the ocher, which has been previously prepared, with the crude oil to be refined, the mucilage, gummy, and coloring matters are absorbed by the ocher, which with the impurities settles to the bottom, leaving the supernatant oil clear and refined. The refining agent is first prepared by heating, so as to drive off all moisture, which is an essential part of the operation. In some cases

the ocher is dried and finely pulverized and used in the proportion of 12 parts of oil to 1 part of ocher. The combined oil and ocher is then briskly agitated for about 35 or 40 minutes, when the refining will be completed. Naturally, these proportions vary with the amount of impurities the oil contains, the same as with other methods of working.

The oil is separated from the ocher by the use of the filter press or allowed to remain in the bleaching tank for 24 hours. In the latter case the clear supernatant oil is drawn off, leaving only the settlings to be filtered. These settlings when removed from the filter press is a stiff paste of ocher mixed with a small amount of oil and all the impurities.

19. The inventor further claims that by a combination of refining or purifying agents, rancid oil may be successfully reclaimed by his process. For oils that have undergone more or less change by oxidation and are heavily charged with free fatty acids and other impurities, the ocher is reduced to a fine powder and 10 per cent. of the amount to be used is taken and mixed with finely powdered caustic soda or borax, the amount taken being equal to from .5 to 1.5 per cent. of the weight of oil taken. This mixture is then added to the oil to be refined and the whole agitated thoroughly for 10 or 15 minutes. A mixture of salt or chloride of calcium in saturated solution, equal to twice the weight of the first mixture, is after this period added to the oil and the agitation continued for 10 or 15 minutes longer. The remaining portion of the ocher necessary to complete the refining is now added to the mixture and the agitation continued for a like period, the temperature of the oil being maintained at about 90° F. The oil is then passed through the filter press to remove the refining agents and the impurities. The chief objection to the use of this combination process, or to the use of ocher itself, is the shrinkage in refining, owing to the necessary saturation of the ocher with the oil. But with rancid oils it might be made very serviceable.

20. Special Treatments and Precautions in Crude-Oil Refining.—In the refining of crude cottonseed oils, many of the details vary in different refineries, and these are also largely influenced by the desired character and destination of the refined oil. Oils that are destined for food purposes cannot, or, more truly, should not, be treated as oils intended for industrial purposes. For the former purposes, as low a temperature only as practicable should be given such oils, as the whole object is to make them as neutral and as bland as possible, with no rank or strong odor, which is always produced in oils when a high temperature is used in working. In the finishing tank care must be taken to keep the oil intended for edible purposes at as low a temperature as is compatible with finishing the product. This will be more fully described under the heading "Butter Oil."

21. An oil unduly heated in the initial process of refining invariably requires more heat in the last processes to brighten it, and such heat is fatal to its essential characteristics as a table oil.

When cottonseed oils are refined for industrial purposes, before the caustic-soda solution is dropped into them, they may be heated as high as 110° F. After the cold lye has been added and the proper breaking of the oil observed, the supply of caustic solution is shut off and the mass in the refining kettle heated, by means of the closed steam pipes, to a temperature of 130° F., the agitation being continued during this heating. At this temperature the steam is shut off, the agitation stopped, and the oil allowed to settle as usual. It is then drawn into the rectifying, or refining, tank and heated in order to impart to the oil the necessary brightness. This oil may be heated to 250° or 285° F., but no higher. By this method the loss in refining the oil is lessened, but the oil is of inferior flavor and taste. In other respects it is similar to oils refined by the lower temperatures.

22. Steaming the oil is resorted to in some refineries both before and after the clarification. For this operation

the oil is pumped through the filter press into another clarifier and free steam is injected into the oil. In order to obviate too great a condensation of steam, the oil is at the same time heated by the closed steam coils. As live steam does not injure oil, the heat may be raised with impunity to 280° or 290° F. This process has the advantage of washing the oil and at the same time bleaching it, first by the oxygen injected with the steam, and second by the fact that the coloring matter of the oil, being held simply in mechanical suspension, is forced out and, when the operation of steaming is finished, drops to the bottom of the tank with the condensed water from the steam.

This process must not be carried on too long, as oil so treated has a tendency to become more or less oxidized, and therefore strong and rank smelling. But properly handled, it is in many cases, especially with poor-grade oils, a great improvement. A steaming not exceeding 10 or 15 minutes will effect this. But in this procedure, as with all cottonseed oils, the class of oil under treatment governs these conditions, and practical experience is the only guide that can be safely followed. Another advantage is the fact that the entrance of the caustic-soda solution may be stopped when the oil is yet reddish in color and the remaining color driven out by steam. It also effectually removes all traces of alkali that may remain after the caustic-soda treatment. The oil after being steamed goes to the rectifier for drying out, as usual.

The steaming of crude cottonseed oil, prior to clarification, is resorted to more particularly with inferior or unclean oils. The free steam dissolves the mucilage of the oil and facilitates its precipitation, as well as many of the impurities that it gathers, especially from tanks and barrels, when received in those receptacles. In many instances, oils are washed with warm water after clarification and are afterwards drawn off into the rectifying, or finishing, tank.

23. Refining Cottonseed Oil by Brine and Caustic Soda.—A patent was issued a few years ago for a process

of refining cottonseed oil by means of brine and caustic soda. The method is said to be very satisfactory. It consists in agitating the crude cottonseed oil in a tank in which is mixed a suitable proportion of salt water. When a thorough mixture is obtained, the usual caustic-soda solution is added until the oil is broken. The agitation is then stopped and warm water sprinkled over the oil. The clear oil rises to the top, the coloring matter subsiding with the water. The temperature during the operation is maintained at 80° F.

24. Loss, or Shrinkage, in Refining.—This is wholly dependent on the grade of the crude oil operated on. While the usual and normal loss in refining will average in this country about 8 or 10 per cent., it may reach as low as 3 per cent., as has been repeatedly demonstrated. Again, with a very poor oil the shrinkage will reach as high as 15 per cent. or more, but the latter figure is rather unusual with the average grade of crude oils. The oils from different sections of the South, while apparently of the same grade, produce very different results as to the loss in the usual methods of refining. The quality of oil also varies in different seasons from seed obtained from the same sources.

25. Each batch of crude oil to be refined presents in a way an original proposition, previous experience with other oils being no help or guide as directly applied to the lot under consideration. Individual lots must have applied to them individual treatment. For this purpose, the color test described later is determined for each separate batch of crude oil and the treatment determined from the data obtained from it.

26. Cottonseed Fouts and Their Treatment.—After withdrawing all the oil obtainable from the refining tank, there remains in the bottom a light brownish material consisting of soap, mucilaginous matter, coloring matter, and other impurities mixed with more or less of the refined cottonseed oil. This is technically known as cottonseed fouts. They are withdrawn into the soap-stock tank *D*, Fig. 1, immediately below the refining kettle, through the large valve *c*.

The foots are slowly heated by means of the closed steam coil in the kettle and the oil is removed, as far as practicable, and mixed with the crude oil for subsequent refining operations. The stock is then cooked and barreled while in a fluid condition and sold under the name of cottonseed foots to soap manufacturers, on the basis of the contained fatty acids.

27. In many establishments the foots are made into soap stock, and subsequently soap, without the intermediate expense of barreling. Cottonseed foots when fresh are of a brownish color, but they rapidly become black upon exposure to the air. In some plants, a crude system of pressing is resorted to for obtaining all the oil possible from the foots, which is an economical procedure, as there is always a wide difference in value between the foots and the oil. By the usual procedure, the free oil remaining in the foots may be saponified, and thus a homogeneous soap obtained. This is known in the trade as **cottonseed soap stock**.

*** 28. Preparation of the Caustic-Soda Solution for Refining.**—The dissolving of caustic soda used in the refining is a simple operation, yet much time and annoyance may be saved by having the lye tank properly constructed. If elevated above the level of the floor, it should be conveniently near the refining tank and always at a higher level to allow the caustic solution to flow into the oil by gravity.

29. The lye tank should be provided with a perforated-metal false bottom, to support the broken lumps of caustic soda. This greatly facilitates solution by causing a natural circulation of the liquors of different densities, and by this, fresh, lighter liquors are constantly dissolving the caustic. The saturated liquor, owing to its greater density, sinks to the bottom of the tank; where the false bottom is not in the lye tank, mechanical agitation must be resorted to for effecting a rapid solution of the alkali. The disadvantage of allowing the caustic soda to dissolve slowly is that it is very liable to become carbonated, thus reducing

its effectiveness in refining where a full caustic effect is required. A carbonated caustic-soda solution is an uncertain factor with which to work in refining. The solution of alkali should always be kept tightly covered and kept at the degree of strength best adapted for refining the usual run of oils; the degree of strength usually selected is 20° Baumé for the stock tank.

In some refineries the stock of lye in the tank is kept in any convenient place and the amount of solution required for each batch of oil pumped from the stock tank into the elevated lye tank above the refining kettle. Many refineries have the dissolving tank on the ground floor, which is a very convenient arrangement.

30. Caustic soda comes on the market in a great variety of grades and is always sold on a basis of 60 per cent. of sodium oxide, Na_2O . The common impurities occurring in caustic soda are sodium chloride, sodium carbonate, and sodium sulphate. It is of great importance to the refiner to know the actual caustic strength of the soda and the amount of the impurities in it. In the preparation of the lye, the latter salts reduce the active value of the solution for the indicated specific gravity or Baumé. In other words, a lye of 20° Baumé, containing a notable amount of impurities in the form of carbonate, etc., would not perform the work in refining the oil that a lye of the same Baumé prepared from a caustic soda of high grade, free from notable amounts of these salts, would do.

As the reduction in the active value (saponifying power) is least in the highest grades and greatest in the lower grades, as a result of the larger proportion of impurities present, it is not only uneconomical to use a low grade of caustic soda in refining, but it often leads to uncertain results.

31. The table given shows to what extent an indicated Baumé reading may show a varying strength of actual caustic soda present in a lye prepared from different grades of ordinary merchantable caustic soda. It is assumed that all the

alkali present is entirely caustic soda, and while the results are slightly higher than would be found perhaps in actual practice, the table is of value and sufficiently accurate for technical purposes. The oil refiner may conveniently consult the accompanying table in preparing his lyes for refining crude oils.

**PERCENTAGE OF SODIUM HYDRATE $NaOH$ IN LYES
OF DIFFERENT DENSITIES MADE FROM
CAUSTIC OF VARIOUS GRADES**

Specific Gravity	Degrees Baumé	77½ Per Cent. $NaOH$	76 Per Cent. $NaOH$	74 Per Cent. $NaOH$	72 Per Cent. $NaOH$	70 Per Cent. $NaOH$	60 Per Cent. $NaOH$
1.075	10	6.55	6.42	6.25	6.08	5.91	5.06
1.091	12	8.00	7.84	7.63	7.43	7.22	6.19
1.116	15	10.06	9.86	9.60	9.34	9.08	7.78
1.142	18	12.64	12.40	12.07	11.74	11.41	9.78
1.162	20	14.37	14.09	13.72	13.35	12.97	11.12
1.180	22	15.91	15.61	15.19	14.78	14.36	12.31
1.210	25	18.58	18.23	17.74	17.27	16.78	14.38
1.241	28	21.42	20.99	20.44	19.89	19.33	16.57
1.263	30	23.67	23.21	22.60	21.99	21.37	18.32
1.320	35	28.83	28.28	27.53	26.79	26.04	22.31

32. For the reasons explained, caustic sodas of 74 and 76 per cent. are superseding the lower grades in the refining of crude cottonseed oils. The caustic is best melted by a jet of steam until the solution is of a density of about 35° Baumé. The solution is then allowed to settle and, if necessary, drawn off into another vat.

The stock-lye tank is usually kept at a strength of 20° Baumé and reduced or strengthened as occasion demands. For making a butter oil, the strength is reduced to 10° or 12° Baumé. For ordinary oils, a strength of 20° Baumé is used, while for poor and bad oils a 25° Baumé may be used. The volume of lye used varies from 5 to 20 per cent. of that of the oil, according to the quality of the crude cottonseed oil.

THE MANUFACTURE OF VARIOUS GRADES OF REFINED COTTONSEED OIL

33. Butter Oil.—That grade of oil known in the trade as **butter oil** is a very choice summer-yellow cottonseed oil made from selected seed, as a rule, and treated somewhat differently in refining from prime summer-yellow oil. Butter oil usually can be made only from crude oils testing not over 1 per cent. in free fatty acids; the crude product must be handled very carefully, regarding the temperatures to which it is subjected, in every operation from the start to the finish. With this oil it is imperative to have every tank holding it scrupulously clean. Although this condition should prevail with every grade of oil, a high-grade butter oil cannot be obtained unless these conditions are rigidly observed, extending this condition also to the barrels into which the oil is drawn for shipment.

In the manufacture of a butter oil, it must meet the trade requirements as to color, taste, flavor, etc., together with a minimum loss in refining to the manufacturer. The process is carried out as described in Arts. 12, 13, and 14, with the following modifications: The crude oil having been selected for manipulation is freed as far as possible from the ever-present impurities by subsidence, decantation, or filtration. An oil that has been settled for several days or weeks has usually all mealy matter, mucilage, etc., wholly settled from it and, being free from sediment, may be pumped directly to the refining tank. Where rapid manipulation of freshly made crude oil is desired, recourse must be had to the filter press to eliminate the impurities.

34. The crude oil is warmed to 75° F. and the agitator started as usual. The caustic-soda solution for refining should be reduced to a strength of about 10° Baumé and in no case exceed 12° Baumé. The oil is agitated in the usual manner and the lye added as previously described. Care should be taken not to heat this crude oil in the refining tank above 105° F. After the oil has been refined and well settled in the usual manner, it is drawn off from the foots

into a thoroughly clean tank and there washed free from all traces of alkali. The butter oil is then freed from moisture by heating and blowing, the temperature in the finishing tank in no case exceeding 120° F., after which the oil is pumped through the filter press to clear and brighten it. The filtered oil, which should be of a light lemon-yellow color and free from all undesirable odor and flavor, is sent to the storage tank for barreling after it has regained its normal temperature. Butter oil should be as neutral as possible in all its characteristics.

35. Yellow Cottonseed Oil.—This grade, which forms the staple of cottonseed oils, is the product of the refined crude oil. It is sometimes heated in a jacketed tank to remove the strong odor and flavor when present in a marked degree. The dried and filtered oil is slowly heated to a temperature of 370° or 400° F. and very lightly blown in some cases, for 3 or 4 hours. This treatment is very beneficial to some oils, but it is disadvantageous with others. Frequently the yellow oil is simply heated to 400° F. and held there for 4 hours, when most of the strong and persistent odor of the cottonseed will be eliminated. Oils treated in this way are liable to have fixed in them the yellow coloring matter, rendering them incapable of being made into a water-white cottonseed oil. Such oil, however, is suitable for use in articles where their yellow color is not objectionable, such as in oleomargarine, "cotto" compounds, etc. Care must be taken not to blow the oil too strongly or for too long a period.

Yellow cottonseed oil may be considered as the raw material for obtaining the various other grades of cottonseed oil, as may be seen by the chart of the resolution of cottonseed in *Cottonseed Oil and Products*, Part 1.

36. White Cottonseed Oil.—This is made by bleaching choice yellow cottonseed oil. It is necessary in the bleaching process to have the oil in question thoroughly free from all moisture. This is a most essential point, as no satisfactory

bleach can be obtained without this condition being present in the cottonseed oil.

37. Bleaching Apparatus.—The apparatus necessary for bleaching by means of the usual fullers' earth is shown in Fig. 2, in which *A* is a cone-bottomed boiler-iron or steel tank, provided with the heating coils *c, c*, the valve *v* regulating the steam, the blower pipe *x*, the siphon, or draw-off pipe *d*, with a swivel joint, and the cock *m* in the apex of the cone. The blower pipe is terminated by a circle of perforated iron pipe of slightly smaller diameter than the blower pipe *x* in the tank *A*. The area of these perforations must in the aggregate be equal to that of the blower pipe *x*, and no more, to secure the full benefit of the air pressure from the blower.

The filter press *R* is in close proximity to the bleaching tank *A*, but protected in any suitable manner from the flying dust occasioned by the fullers' earth being added to the oil, so that the dust will not contaminate the finished, filtered oil. In most cases in the oil refinery, the filter press is placed in an adjoining room to afford the desired protection from the flying fine particles of the fullers' earth.

38. The bleaching is carried out in the following manner: The yellow oil having been run into the tank *A* through the pipe *b*, the blower is started and the oil heated and blown, if circumstances require, until the moisture has been entirely eliminated. This is ascertained in the manner described in Art 13. The temperature may be carried to a high point within limits, as the hotter the oil, the better the fullers' earth will effect the bleach. A good temperature for bleaching yellow oil is from 190° to 210° F. The heating and blowing being concurrent, when the oil reaches the temperature above mentioned, all the moisture has been expelled. The fullers' earth is now added in sufficient quantity to accomplish the bleach; the manner of approximately ascertaining the quantity of this material required will be described later. The valve *o*, Fig. 2, is opened after the oil has been agitated for a few minutes, and the oil and

clay pumped through *e* to the filter press. The latter has always a connection leading from its trough back to the bleaching tank, so that the first portions of the oil may be returned for a more perfect bleaching. The circuit of the oil from the bleaching tank, through the filter press, back to the tank is maintained until a sample of it in the test glass is seen to be of the required white color.

39. The coloring matter having been entirely removed by the bleaching process, the oil is directed through *j*, by changing the three-way cock *g*, to the storage tank *H*, where it is allowed to remain until it has cooled to the room temperature before being run into barrels. This is necessary to obtain the oil in packages in a clear, sparkling condition. If the oil is drawn into barrels while it is hot, the result will be a cloudy oil, materially deteriorating the market value of an otherwise perfect oil. During the pumping of the bleaching oil from the tank, the agitation is continued on the material to prevent the fullers' earth settling to the bottom of the tank, thereby preventing its exerting its full bleaching action on the oil.

40. After all the material has been pumped from the bleaching tank, the air blower is shut off from the latter and turned on to the filter press to remove the bleached oil held between the pleats and in the cloths. The press is then blown until no more oil escapes from the outlets. If the press is not desired for immediate use, steam is blown through it to steam out all oil held by the clay and the cloths. This oil should not be mixed with the previously bleached material, but saved by itself and treated in subsequent operations. The cloths are then dried by blowing the air from the compressor through the press.

If the filter press is to be used within 24 hours for the same purpose, the steaming operation is not performed, but after blowing allowed to remain without further change, provided the quantity of fullers' earth used in the bleaching has not filled up the space between the filter-press plates.

Where a large quantity of clay has been used, it is necessary after the oil has been steamed out and the cloths dried to remove the dried clay from the latter by scraping them with a dull-edged scraper. The same cloths may then be used without removing and washing, which, however, must be done in any case after the cloths have been used on the filter press three or four times.

41. In bleaching yellow cottonseed oils, it is always advisable to perform a test on a laboratory scale to ascertain its bleaching qualities. It will frequently be found that some yellow oils cannot be bleached to a water-white color and also in some cases that such an excessive amount of fullers' earth is required for this that it is not profitable to attempt to bleach them. Some yellow cottonseed oils are not susceptible of bleaching to any degree, and hence the advisability of always making the preliminary test on a small scale is apparent. Coincident with this is the quality of the fullers' earth used for bleaching purposes, which is a very important feature in the bleaching of cottonseed oil and is discussed later.

42. In bleaching yellow cottonseed oil in the tank, it is always advisable to use as low a temperature on the oil as will effect the bleach. The lower the temperature at which it is bleached, the better will be the flavor of the oil produced. Certain yellow oils bleach at a much lower temperature than others of the same grade, hence no rule applicable to all oils can be given. If too high temperatures are used in bleaching yellow oils, they are liable to be burned and then it is not possible to bleach them. An excess of fullers' earth is always to be avoided in this process, as all added over and above the quantity required to produce the white oil acts in a detrimental manner by imparting to the bleached product an objectionable taste and flavor known as an *earthy* or *clay* taste or odor.

There are two grades of white cottonseed oil, prime white and off; the difference consists generally in the flavor and

color. Winter white oil and summer white are made, the difference between them consisting solely in the cold test, explained later.

43. Preliminary Bleaching Test.—In testing the bleaching qualities of a yellow cottonseed oil, a 4-ounce sample bottle is filled with a known weight of the oil, 100 grams being a convenient quantity. The bottle and oil are heated on a steam bath to 180° F., a quantity of fullers' earth equal to the percentage judged to be required is added to the hot oil, the bottle corked, and the clay intimately mixed with the oil by violently shaking the bottle for 3 or 4 minutes. The contents of the bottle is then poured into a folded filter and allowed to run into another bottle. As the first portion of the filtrate is most likely to be more or less cloudy with fine particles of clay, this is poured back upon the filter until the filtered and bleached oil passes through perfectly clear. By observing the color and comparing the oil with the original oil for treatment, the capability of bleaching may be judged, and also the approximate quantity of fullers' earth required to produce a white oil. Should the color not be satisfactory with the first test, a second test is made, increasing the percentage of clay added. In this way a very close approximation may be obtained.

44. As the air agitation in a tank of oil brings every particle of clay into intimate contact with the material, a far better effect is always obtained there than is possible in the bottle test. For this reason it will always be found that about one-quarter less clay will be required to effect the bleach of the oil in the tank than is indicated by the test carried out in the bottle. In the latter, care must be taken to have the oil free from moisture and the bottles perfectly dry before the test is begun.

45. Fullers' Earth.—This is also known in a technical sense as "clay." It is a peculiar form of clay that, unlike ordinary clay, falls to pieces when placed in water. It has

the property of absorbing water to a great extent without becoming pasty. Its most important property is that of absorbing coloring matters from oils. The chemical analysis of fullers' earth gives no clue whatever as to its value as a bleaching agent. Two samples may have practically the same chemical composition and yet differ radically in their physical properties in their effect upon an oil subjected to their action for bleaching. While one kind of fullers' earth may give most excellent bleaching results, the other, with the same chemical composition, may have no bleaching effect whatever. It is the concensus of opinion, confirmed by practical experience and investigation, that the bleaching properties of a fullers' earth are due entirely to its physical structure or condition.

46. In the selection of a bleaching clay, care should be exercised to make a test of the sample on a laboratory scale against a sample of known bleaching power for comparison. Equal weights of the clays for the test are taken and the same weight of oil bleached with the fullers' earth and the results noted. Should a new lot of fullers' earth be superior in its bleaching power to that held as standard, the new clay is then retained as a standard for future purchases. There are many inferior bleaching clays on the market and no large quantities should be purchased without the application of the laboratory test to the sample. The test described for bleaching yellow cottonseed oil may be applied similarly for this purpose also.

English fullers' earths are most largely used for bleaching cottonseed oils, the color of the earth itself exercising no apparent influence upon its bleaching qualities. They are yellowish and bluish, frequently approaching black in color. Of late years, American fullers' earths have come very much into favor, and some of them do as effective work as the foreign article.

A fullers' earth for bleaching should be very fine and the clay itself free from sand and foreign material. The finer the clay, the better are the results that can be obtained

with it. A bleaching clay should be ground fine enough to pass through a 100-mesh sieve. The drier a fullers' earth is, the better is the bleach that can be obtained with it; hence, it is important that this material should always be stored in a dry and, preferably, warm place.

47. Winter Cottonseed Oils.—Most animal and vegetable oils, when chilled, deposit the less readily fusible constituents as stearin. This is especially true of cottonseed oils, both crude and refined. On subjecting the settled portion of the oil to filtration and pressure, the more fluid portion, the olein, may be obtained, practically free from suspended albuminous matters. The impurities are retained to a great extent in the separated stearin. By allowing cottonseed oil to stand in the tanks at a relatively low temperature for some time after refining, the heavier part of the oil containing the stearin settles to the bottom of the tank. This procedure is frequently resorted to as a means of crude separation, as the upper part of the oil, especially in cold weather, may be drawn off and fulfil the requirements of a winter cottonseed oil. The thickened mass remaining in the tanks may then be pressed to obtain the cottonseed stearin, thus effecting a saving of time and much labor in handling.

Oils that remain limpid and clear in a cold temperature (winter weather being in a way understood) are termed **winter oils**, while those that become turbid or partially solidified on chilling are designated **summer oils**.

48. Recovery of Cottonseed Stearin in Making Winter Oils.—The making of winter cottonseed oil is concurrent with the production of cottonseed stearin. The refined oil that is to be resolved into winter oil and cottonseed stearin is allowed to rest without agitation in a cool temperature, about 30° or 35° F., until the solid portion of the oil has subsided. This settling, or chilling, is done in receptacles so arranged that the light, thin portion of the oil, the oleins, may be withdrawn without disturbing to any extent the

sediment underlying them. The time required for the separation of the two component parts depends largely on the individual character of the oil under treatment, a period of 10 to 15 days generally being sufficient for the separation; at times, a much shorter period accomplishes this. The longer the period of subsidence, the more complete the separation and the denser the settled stearin portion will be. The tank in which settling takes place may be an ordinary open-head tank, in which are placed draw-off cocks at various distances on the upright tank, by means of which the supernatant, clear oil may be withdrawn from the underlying thick portion.

49. The chilled mass, containing the cottonseed stearin, with more or less oil, is removed and then subjected to the usual pressing, the material being encased in duck press cloths of close texture. A press suitable for this purpose is shown in Fig. 3.

The thick material is filled into the cloths placed over a wooden mold, in order to make the cakes of a uniform thickness and of a size adapted to the form, and the sides and ends of the cloth folded over it, thus forming a bag containing the material. These bags are about 3 inches thick, 14 inches long, and 7 inches wide; they are then placed in the press in layers, each separated from the other by thin plates of sheet iron. In placing the bags in the press, care should be taken that they are placed a sufficient distance apart from one another to allow for the spreading of the material in pressing, thus avoiding the crowding together of the pressed cakes. A distance of about 3 inches between the bags on the plates will give ample room for the material to spread and press out. The cakes should not be allowed to project beyond the edges of the iron plates, or those portions will be insufficiently pressed. In pressing the molded cakes, the pressure should be very slowly applied, to allow the material to gradually acquire a firm condition. A sudden and quick pressure at any stage of the operation will result in bursting many of the cloths.

50. After the press is filled the material is first allowed to drain without pressure. When the flow of the oil from the natural weight of the layers upon one another ceases, the pressure is gradually applied by the descending plate, frequent periods of rest being permitted for the oil to drain from the stearin in the cakes. The pressing is continued



FIG. 3

until the stearin and oil have been more or less completely separated, which may be seen by the oil ceasing to drain off under the heavy pressure applied. The press, Fig. 3, is provided, by means of pulleys, with a quick motion for closing up the material and for raising the top plate for unloading, and with a very slow motion, which may be regulated to almost any speed, for the pressing proper.

The room in which the press is placed is cooled by usual refrigeration methods, so that no melting of the stearin takes place, as otherwise this would go into the winter oil and cause the latter to show a cloudiness upon being chilled for the cold test. The pressed-out oil is conducted to the usual storage tanks and, if necessary, may be run through the filter press to remove any extraneous impurities. This oil is *winter-pressed cottonseed oil*.

51. The solid portion of the cottonseed oil remaining in the cloths is the true cottonseed stearin. It is a light yellowish fat of a buttery consistency and is nearly neutral in its characteristics. It is usually pressed so that it has a melting point of 38° C. There is no standard for the hardness of this stearin, it being sold usually by sample. The longer and the harder within limits the mixture of stearin and oil is pressed, the harder the resultant stearin will be; and the lower the temperature in chilling and pressing, the freer from stearin the winter oil will be.

The pressed material is removed from the cloths and disposed of to the trade for various purposes, among them being the manufacture of edible products.

52. Wesson Process Oil.—Very recently there has appeared upon the market a very superior vegetable oil designated as *Wesson process oil*, one brand being used as a salad oil and another brand as a cooking oil. This is a very superior cottonseed oil, especially prepared by a secret process, being devoid to a marked degree of the usual characteristic odor and smell of the ordinary oil. It appears like a cold-pressed oil and possesses many characteristics of cottonseed oil prepared in that manner. The chief claims of the manufacturers of this oil are, first, a neutral and palatable flavor and, second, freedom from smoke and odor in frying—points that are not possessed by the ordinary cottonseed oil used for cooking purposes.

53. Oxidized or Blown Cottonseed Oil.—The manufacture of oxidized oils is a comparatively new industry and originated in England. Great hopes have been, and are

still, entertained that by the oxidation of cottonseed oil, a substitute for castor oil, with all its specific purposes, might be obtained. Up to the present time this has not been accomplished, the use of oxidized or "blown" vegetable oils for lubricating purposes not being as general as anticipated. Like many new products, extravagant claims have caused it to be undervalued, but it has, nevertheless, a great many meritorious characteristics.

Oxidized cottonseed oil is very similar chemically to castor oil. Its acetyl number (see *Quantitative Analysis*) is about 80, as against 17 before oxidation, and the oxidized product is far more soluble in alcohol (characteristic of castor oil) than the original untreated oil. Acids corresponding to sulpho-ricinoleic have, however, not yet been obtained from the treated product. While this oil may be used, both alone and mixed, for lubricating machines of great power, such as hydraulic presses, it has not succeeded in replacing castor oil in soap making, as it will make only dark soaps possessing far different properties from those made from castor oil. Oxidized oils are most suitable and best adapted for making india-rubber substitutes by the action of sulphur or chloride of sulphur. The blown cottonseed oils have many uses that are held as trade secrets, concerning which no data can be obtained.

54. The process of oxidizing is carried out in the following manner: Only winter cottonseed oil, free from stearin, is used for this purpose. The oil is placed in a tank provided with the means of heating, blowing, and agitation; the bleaching tank *A* shown by Fig. 2 is suitable for this purpose. The oil is heated without blowing to 100° C.; at this point the air, in finely distributed jets, is sent through the oil from the perforated pipe in the bottom of the tank. The first effect is to lower the temperature of the oil, but it soon rises again to a point much higher than the initial temperature. When the temperature has reached 130° C., the blast is shut off and the viscosity of the oil tested. If a sufficiently high viscosity has not been reached through the

first blowing, the oil is allowed to fall to 100° C. again, when the air is again blown through it until the temperature of 130° C. is obtained. This oil is again tested to ascertain its viscosity, which may be compared with a standard sample or with castor oil. If, through the two blowings the oil has not acquired the desired consistency, the operations of successive blowings and cooling are performed until the result is accomplished.

55. The duration of the operation may be materially shortened by adding to the oil from $\frac{1}{8}$ to $\frac{1}{4}$ per cent. of its weight of a drier. A manganese salt is usually selected for this purpose, the resinate or borate generally being employed. The drier is added to the oil just before starting the air blast, when the oil is at 100° C.

In the operation of blowing and consequent oxidation, care must be exercised not to allow the temperature to rise above 130° C., or the oil will become permanently darkened and thereby greatly deteriorated in value for sale purposes. If the high temperature is maintained too long, the oil assumes a tawny, dirty color, and in addition such overheated oils are very difficult to mix with mineral oils. Oxidized cottonseed oil resembles closely in appearance ordinary castor oils, but is always more or less sticky.

The operation may be performed with oxygen or ozone, but for this purpose the necessary apparatus and plan for generating these must be added to the ordinary equipment.

COOPERAGE

56. General Remarks.—In the cottonseed oil industry *cooperage* is one of the most important features tending to profit or loss. The profits in some seasons are so small that losses arising from the use of poor oil barrels may completely obliterate it. It is a most excellent investment to use for shipping oil in barrels only the best, well-seasoned packages.

Barrels made from cross-grain or sappy, green wood should never be used for shipping cottonseed oil. Staves from the former are very easily cracked in handling, thereby allowing a constant leakage in transit or storage. When green or sappy wood is used, the drying out causes the wood to shrink, thus opening the cracks between the staves and causing such packages to be useless until recoopered, or, as it is technically termed, *trimmed*.

57. Trimming Barrels.—This consists in removing each head of the barrel and then inserting between each stave pieces of dried flag. The latter being of a dry, spongy nature completely fills the cracks between the staves and accommodates itself to the varying pressure on the iron hoops when driven as tightly as possible. When all the interstices between the staves around the heads are filled with flag, the heads are again put in and the package made as tight as possible by driving the iron hoops until they will stand no more strain.

The oil barrels before filling should be lined with some impervious material, such as glue or paraffin.

58. Lining Barrels.—When it is desired to line a barrel with glue, about 2 or 3 quarts of a heavy, hot, glue solution is poured through the bung hole of the barrel which is ready to be filled with oil. When the bung is replaced, the barrel is rolled and turned over in every direction so as to thoroughly coat every part of the inside surface. The barrel is then placed, with the bung out, over a trough and allowed to drain free of the excess of glue solution, which is collected and used over again. By this treatment all pin holes (worm holes) are stopped up and any cracked staves coated so that they will not leak oil. It is customary to line the barrels in the afternoon so that they may dry over night and be ready for filling the next morning.

In using paraffin for lining oil barrels, care should be exercised to choose a paraffin of comparatively low melting point. Material of this nature having a melting point of

126° F. is suitable for this purpose. When paraffin of a high melting point, for instance, 136° F., is used for lining, when cold, it is apt to crack when the barrels are subjected to falls or rough handling.

Silicate of soda has been used for lining oil barrels, but it has fallen into disfavor, owing to the liability of some of the oil to become partially saponified when placed in barrels in which the silicate of soda has not had time to thoroughly dry hard. Oil placed in such packages becomes milky and very cloudy and consequently much deteriorated in value.

The labor attached to the lining process is much lessened by selecting in the first place barrels made from sound and well-seasoned wood. In fact, the best and most economical investment in every way is to use for oil barrels only the best possible cooperage obtainable.

APPLICATION OF THE FILTER PRESS TO THE COTTONSEED-OIL INDUSTRY

59. General Remarks.—The use of the filter press in cottonseed-oil milling is of quite recent date. Even at the present time there are many mills that are not equipped with this important piece of apparatus, by means of which large quantities of material may be handled and manipulated rapidly and inexpensively. Why its use is objected to by some oil millers is for reasons known only to themselves. In every industry where filtration or clarification is a requirement in the various manufacturing processes, the use of the filter press has proved beyond all question its value as an economical factor.

In the clarification of vegetable and animal oils, as a part of the refining process, the filter press is accorded a prominent place. So effectually does it prosecute the work of refining, with especial reference to the elimination of solid and generally extraneous substances, that all previous methods for the accomplishment of the same purpose have become obsolete in this country. The requirements of this

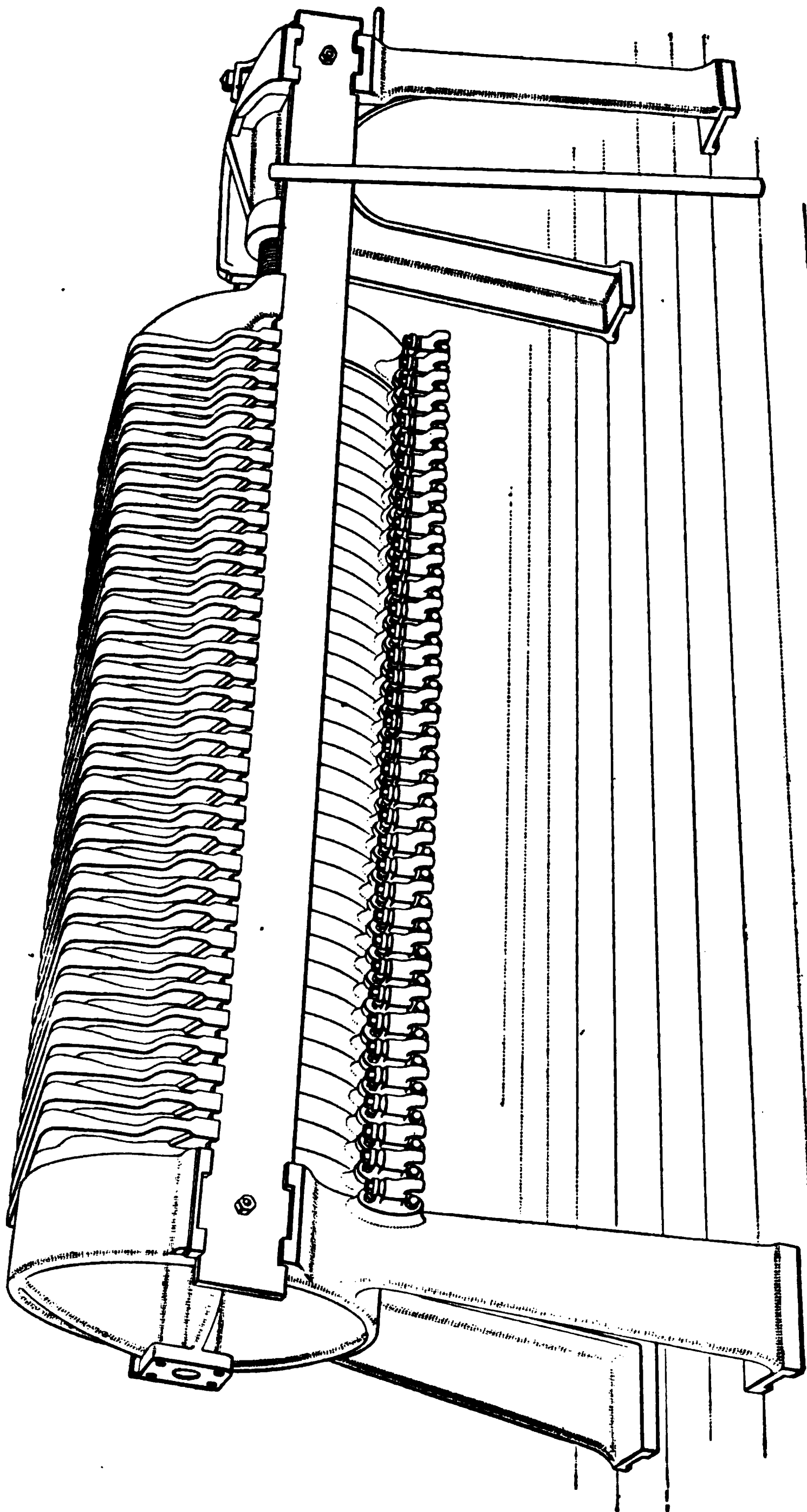


FIG. 4

age for a superior and more quickly prepared finished product have become too urgent to permit of the continued use of the old-fashioned settling tank and gravitation filter.

60. In the cottonseed-oil industry the filter press is of great economical importance in many ways. By its use it renders practicable the transformation of the crude oil into a merchantable product within a few hours after its extraction from the crushed cottonseed. Previous to its introduction into this industry such a proceeding was impossible of attainment, owing to the protracted and long period of rest required for the precipitation of the flocs and fine vegetable matter in the production of a clarified oil. It is a well-known fact that oil drawn from the settling tanks, such as were used previous to the installation of the filter press, in many cases would frequently show some particles of solid and fine extraneous matter after months of repose. This proving such a serious drawback, the inventive ingenuity of refiners brought forth a number of methods having for their object the separation of the solid impurities from the oil in which they remained suspended.

While slight improvements were effected from time to time, defective work under the systems that then prevailed proved detrimental to the trade in general. The gummy or mucilaginous substances, the ever-present accompaniments of expressed cottonseed oil, are naturally very slow of subsidence and precipitation. Their complete removal becomes absolutely necessary in obtaining a clarified, or refined, cottonseed oil. This is effected in a thorough and very rapid manner by means of the modern filter press.

61. Filter Press.—The filter press shown in Fig. 4 is one of the most modern type, and has lately been introduced into several of the largest cottonseed-oil refineries, as being especially adapted for this class of work. One of the improvements over the usual style of press is applied to the filter plates, which, instead of being simply

grooved, are studded all over the surface with small, flat-topped pyramids; a section of such plate is shown in



FIG. 5

Fig. 5. These pyramids support the filter cloth, and at the same time afford an effective passage for the filtrate to pass away, making a marked improvement in the rate of filtration. The equipment, with regard to the filtering medium, is described later; a single plate of this press is shown in Fig. 6.

FIG. 6

The cloths having been adjusted on all the press plates, the latter are forced together by a follower, which is actuated by a screw operated by a lever wheel. A very recent device for closing the press is the ratchet lever. This is of great efficiency for the rapid closing and opening of the filter press, and effects a great saving of time and labor. It is constructed on the principle of the ordinary ratchet gear and is operated by the oscillation of the lever. The plates of the press are recessed, so that a chamber is formed between each two plates. The concave surfaces of these are provided with the pyramid

surfaces described, for the drainage of the filtered oil, which passes through the opening in the bottom of the plate.

62. In effecting the filtration of the oil, the material is pumped into the press, and, passing through the cloths, courses downwards through the interstices in the plates, through the outlet, into the receptacle beneath, a perfectly clarified product, the residue—the mealy and mucilaginous matter in the case of crude cottonseed oil and the fullers' earth in the case of bleached oils—being retained in the chambers of the press by the filter cloths. The capacities of filter presses vary with their size, some being capable of handling 40 tons of oil daily.

The construction of the filter press combines the advantages of the largest possible filtering surface in the smallest possible space; the facility of forcing the material through the filtering medium by the most suitable pressure from a slight hydrostatic pressure to a working pressure of 200 pounds per square inch; the ease and facility with which the press can be handled; and the rapid and complete separation of the oil from the residuum and suspended matter. Filter presses are made both of square and round pattern, to suit individual preference, as the shape is wholly a matter of working convenience.

63. It is often desirable to filter a small quantity of oil, such as would fill only part of the filter press. In this case the press has inserted at any convenient part a solid plate without the usual center feed hole and outlet. This plate, known as a *dummy plate*, is shown in Fig. 7.

It is used where less than the full capacity

FIG. 7

of the press is wanted by removing one or two plates and inserting it to cut off the flow. For instance, if only five chambers of the press are wanted, the sixth and seventh plates are removed and the dummy plate inserted, making it practically a perfect working press of five chambers. This is found very serviceable in manipulating small, experimental lots of oil.

64. Filtration.—The rapidity with which a liquid is separated from a solid during filtration depends primarily on the force with which it is urged through the filtering medium and the resistance with which the medium opposes the flow. An increase of force in urging filtration results in an increase of the quantity of liquid filtered during a given time. For this reason, in technical working, means are adopted, usually by the pump, to force the liquid to be filtered through the filtering medium. This increased flow, however, is not a universal rule, as in some cases an increase of pressure, particularly when it is sudden or rapidly applied, actually diminishes the rate of filtration. For every substance, and oil in particular, there is a certain rate of flow, and hence a corresponding pressure, beyond which no increased useful effect results, corresponding with the increased force applied. A little consideration will show why this is so. Coincident with an increased rate of flow there is an increased resistance at the point of filtration, because the real filtering medium is not the particular fabric or surface upon which the solid matters are retained, but the aggregated solid matter itself, which is built up or deposited upon this surface. Hence, the more volume of flow of filtrate, the more solid matter is deposited; the thicker the filtering bed becomes in a given time and the closer the particles deposited are driven together under the greater force, and thus, being more firmly compressed, the greater is the resistance offered the passage of the liquid. It is thus apparent that to secure the most favorable permeable condition of the substance deposited there must be a limit not only to the rate of deposition, but also to the

thickness of the deposit, beyond which useful filtration practically ceases.

65. With crude cottonseed oil, with the gummy and somewhat flocculent substances present, the thickness limiting the useful rate of filtration is likely to be soon reached. In this case, any increased pressure over that which is necessary to filter the body of the oil retards the rate of filtration and will, if continued, ultimately so close the pores of the deposit that filtration will entirely cease.

In applying the pressure in filtering oils, the pump should be started very slowly and the action gradually increased until the maximum pressure effecting the best results from the filter press is obtained. This applies with equal force to both the filtration of crude cottonseed oils and to the bleaching of yellow oils.

66. Filter-Press Cloth.—The filter-press cloth used in the filtering of oils is an important factor in producing good, clear, sparkling oils. No matter how good the filter press itself may be, if it is not supplemented by a good filtering medium the effectiveness of the whole process of filtration is to a great measure nullified. In many of the mills too little attention is given to this important consideration.

The cloth ordinarily used in filtering cottonseed oils is of closely woven texture, more or less elastic and very pliable. It closely resembles duck, but is woven differently; it is known as *chain cloth* from the appearance and style of weave. This material answers most requirements in the oil refinery and it is of moderate cost.

The best material, however, for the filtration of these oils is the press cloth known technically as *lamb skin*. This material is a closely woven fabric having 450 picks, or threads, to the linear inch. One side of the cloth is smooth, the other having a cotton-flannel appearance. The cloth is placed on the filter-press plate with the woolly side to the flow of the oil, being protected from being clogged and stopped up by another cloth placed over it, a *twill* cloth.

The latter is more or less like the ordinary press cloth, but not quite so closely woven. This combination of "lamb and twill" is for cottonseed oils the ideal filtering medium. The reason for its lack of general adoption being its rather high cost.

THE VARIOUS INDUSTRIAL USES OF COTTONSEED OIL

67. General Remarks.—The industrial uses of cottonseed oil are, as has already been mentioned, very numerous and diversified. Its use is constantly being extended to new fields and there seems to be no limit apparent in its wide application, as evidenced by the numerous inventions in which cottonseed oil plays a part. Refined cottonseed oil may be used for almost every purpose to which oil is put except for household illumination and lubrication. It is not practicable, however, to give here an extended account of every use in the arts and manufactures to which cottonseed oil is applicable or adapted, but some of the most important will be described and others indicated and briefly touched upon. The use of cottonseed oil in the manufacture of edible compounds, such as lard and cotton compounds and butterine, will be found described elsewhere.

68. Soap Stock.—Cottonseed oil is the purest form of soap stock that comes upon the market and, as the rules of the various boards of trade, etc., prescribe exactly what its condition and qualifications must be, it is thus always uniform in its characteristics as adapted especially for the soap manufacturer. The oil is made into various grades of soap, from low-grade laundry soaps and washing powders to high-grade toilet soaps.

69. For Cleaning and Bleaching.—Cottonseed oil free from mineral oil or wax is used by bleachers of cloth in a somewhat novel manner. If the goods are found to contain some off-color places or are spotted in any way, some cottonseed oil is painted over the spots by means of a

brush or sponge. A second treatment of the goods in the scouring and bleaching will then remove the off-color places and leave the goods an even white. The oil impregnates the fiber of the goods, softens the discoloring matter, and renders it easy to remove.

70. Culinary Uses.—Summer yellow oil is used largely in culinary operations, being especially adapted for certain processes of cooking. The use of cottonseed oil for this purpose has grown rapidly during the last few years and is still growing. Winter yellow oil finds extensive use both under its own name and also as “olive” oil for salad oils.

71. Pharmaceutical Uses.—For pharmaceutical purposes cottonseed oil finds extensive use as a base for liniments, ointments, and similar preparations in place of olive oil, which a few years ago was used for these purposes. It is an official preparation of the United States pharmacopœia.

72. Illuminating Uses.—For miners’ lamps, the white cottonseed oil is largely used. An Ohio law provides that cottonseed oil, pure lard oil, or their equivalent, only must be used in mines in that State. Although cottonseed oil itself cannot be used in lamps with chimneys, by mixing it with kerosene oil in varying proportions, an excellent oil, known to railroads as signal oil, is made. A suitable mixture for this is 50 parts of kerosene oil to 50 parts of white cottonseed oil. This mixture is burned in brakemen’s lanterns, etc.

73. Substitutes for Linseed Oil.—A very recent claim is made by an inventor in New Orleans that cottonseed oil is capable of being degummed so successfully that it is practically the equal of linseed oil for uses in painting, etc. The process is at present kept secret by the discoverer, and should it prove all that is claimed for it, a further wide utilization of cottonseed oil is thus apparent, which hitherto it has not obtained. In addition to rendering the oil siccative, the gum that is extracted is claimed to be valuable for many purposes, among them being painter’s or gold-size cements and other adhesive purposes. The degummed oil when

submitted to a further process is rendered as clear as water.

74. The following formula gives another substitute for linseed oil: 105 pounds of light Burgundy pitch is melted and mixed with $54\frac{1}{2}$ pounds of raw cottonseed oil and 10 pounds of thick Dutch stand oil, both having been previously heated to 80° C.; 70 pounds of petroleum is now added and the whole heated. When cold, $\frac{1}{2}$ pound of tincture of valerian is added. If the raw cottonseed oil is previously heated with 3 per cent. of litharge, the final product will behave as a varnish. $3\frac{1}{2}$ ounces of resinate of manganese and half that quantity of turpentine answer better than the litharge.

75. Manufacture of Artificial Rubber.—It is said that cottonseed oil has a considerable outlet in the rubber industry. By adding 18 per cent. of crude rubber to this oil, an imitation is formed that it is difficult to detect from genuine rubber. By the action of chloride of sulphur on cottonseed oil, a viscous, sticky compound is obtained, which is the chief component of "factis," or artificial rubber.

76. During the last twenty years the subject of artificial caoutchouc and gutta percha has received considerable attention. The research has been stimulated by the early discovery that isoprene, a hydrocarbon distilled from caoutchouc and oxidized by acids into an elastic mass having the properties of the natural gum, can be obtained also from gutta percha, turpentine, and vegetable oils. The experiments having shown that the artificial gum can be produced, the problem now is how to lessen the cost. The so-called new synthetic gum of M. Eugene Turpin, who is known as the inventor of melinite, is produced by heating vegetable oil to 125° or 250° C. and oxidizing by a current of air or by barium or manganese dioxide, adding as the oil begins to harden 25 per cent., by weight, of colophonous rosin, 25 per cent. of powdered sulphur, 5 or 10 per cent. of spirits of turpentine, and 1 to 2 per cent. of carbon chloride. On reaching the proper pasty consistency, the mass is cooled and is

the artificial caoutchouc. For gutta percha, the formula is varied by omitting the sulphur and spirits of turpentine and greatly increasing the proportion of rosin. The gum is also produced by a wet process, in which the mixture of vegetable oil and rosin is oxidized in boiling water by nitric acid.

77. An artificial product, which for certain purposes can take the place of india rubber and gutta percha, is obtained by mixing oxidizable vegetable oils (linseed oil, cottonseed oil, palm oil, etc.) with tar, creosote, or wood vinegar. Melted or pulverized shellac or shellac solution may, besides, be added. Next, the mixture is treated with diluted nitric acid and a non-viscid, elastic, tough product is obtained, which can be vulcanized.

According to another somewhat modified process, the mass is exposed to the action of nitric acid for a short time only and then heated on plates. The artificial caoutchouc is used either alone or mixed with natural caoutchouc, and is chiefly employed as an insulating material for electric conduits and for waterproofing fabrics.

78. Miscellaneous Uses.—Among other uses of cottonseed oil are baths for tempering steel; in combination with other materials for preserving wood by saturation; for bolt cutting and similar work; for canning fish, and for mixing with higher priced oils for sophistication, to reduce their cost, the exportation of cottonseed oil to olive-producing countries amply evidencing this fact. A curious circumstance bearing on this has recently been revealed by statistics. Some years ago Turkey prohibited by law the admixture of cottonseed oil with olive oil. This action has had a bad effect upon the Turkish oil trade, since it has closed most of the purchasing markets against Turkish oils, whose strong flavor can no longer be masked by the aid of cottonseed oil. Chip oil is a trade term in some localities for a mixture of olive and cottonseed oil. The composition varies with the individual desire of the compounder.

COTTONSEED OIL AND PRODUCTS

(PART 8)

MANUFACTURED AND BY-PRODUCTS

MANUFACTURE OF EDIBLE COMPOUNDS

LARD COMPOUND

1. Lard compound offers probably the largest outlet for cottonseed oil of any article of commerce; it includes such similar edible compounds as cottolene, "cotto" compounds, etc. In former years, owing to the prevailing prejudice against cottonseed oil as an edible material, many subterfuges were resorted to in disposing of these compound lards; at the present time, lard and similar compounds are presented to consumers under their legitimate titles of composition, and the advantages of using vegetable oils in place of animal fats widely advertised.

The proportion of the ingredients used in lard compound varies according to circumstances, such as the season of the year and the locality for which it is designed. The ordinary and usual proportions for lard compound are cottonseed oil, 80 per cent., and oleo stearin, 20 per cent. These are the proportions used in winter or for cool climates. For summer and warm climates the proportions are cottonseed oil, 75 per

cent., and oleo stearin, 25 per cent. These formulas may be, and are, varied by individual manufacturers. In addition to this, other materials are frequently added, such as tallows and, rarely, lard. When oleo stearin is high in price, it is, without detriment to the compound, in part substituted by the less expensive tallow.

2. Selection of Cottonseed Oil.—The cottonseed oil selected for compounding should be as heavy bodied as possible, as the greater the amount of cottonseed stearin it contains, the less proportion of tallow or oleo stearin will be necessary to impart to the compound the desired or required stiffness. Oils which upon standing in a moderately warm place show a deposit of the cotton stearin should be given preference over those that remain clear under the same conditions. The oil selected, prime yellow oil, is bleached with fullers' earth in the manner described previously. It is not necessary in bleaching cottonseed oil for the usual lard compound to have it water white, as the compound usually is of a more or less creamy tint, and in addition to this, the agitation or chilling the compound receives previous to drawing it into packages has the effect of making it whiter. The tallow when employed for this purpose is bleached similar to the cottonseed oil, as is also the oleo stearin. For certain trades, lard in small proportion is mixed with lard compound.

The ingredients are separately bleached and kept so until wanted for mixing, when they are placed in the mixing tank. Here they are kept mixed by suitable agitation and are pumped through the filter press and thence flow to the agitator or over the chilling roll, commonly termed the *lard roll*.

3. Apparatus Used.—The apparatus shown in Fig. 2, *Cottonseed Oil and Products*, Part 2, illustrates the general arrangement suitable for making lard compound. The tank *A* serves for a mixing as well as a bleaching tank. The temperature at which the tallow or oleo stearin is

bleached should not exceed 120° or 130° F., as otherwise a strong and tallowy taste is likely to be imparted to the material. The same general directions previously given relative to bleaching apply to the bleaching of all materials like these. A most important point in bleaching any oil or fat is to have it "dry" or perfectly free from moisture. If such is not the case, when the fullers' earth is added to such material, the earth at once absorbs the moisture present and becomes pasty, and in this condition soon stops the cloths on the filter press from allowing the bleached material to pass through them. This necessitates practically starting the operation again under the right conditions, thus wasting both time and material. In addition to this, oil or fat treated under these unfavorable conditions is more than likely to acquire an earthy or clay taste, an undesirable and most objectionable feature.

4. The **agitator** used for chilling the lard compound is of the usual style of lard agitators. A common form is an open, cylindrical tank of boiler iron, provided on the inside with radial arms fixed to a revolving shaft. The tank used for the agitator has a double shell, through which cold water or brine circulates, thus assisting in rapidly chilling the warm lard compound. The material in the agitator is stirred by the revolving arms until it is almost solid; that is, as stiff as it can be and yet able to be drawn or pulled from the agitator by the pump for filling into the various packages. The lard compound when filled into an ordinary lard pail should be of such a consistency that when the pail is held upside down immediately after drawing, the compound remains in its drawn condition.

5. **Lard Roll.**—Where large quantities of lard compound are made daily, the above method seldom fulfils the requirements of rapid cooling. In such cases, the lard roll, Fig. 1, is used for this purpose. The roll *a* is cast in one piece of iron and varies in length from 4 to 8 feet, being usually 3 feet in diameter. In some cases two rolls are used in connection with each other; one, chilled by ice water, receiving

the hot material, the other finishing the chilling by receiving the partly cooled stock on its brine-chilled roll. The hot material is fed from a tank above the roll, or rolls, at a rate that allows the compound to be perfectly chilled in one revolution of the roll. The speed of the latter is about 10 revolutions per minute. When the chilled compound almost reaches the attached trough *b*, it is removed from the roll by an attached scraper *c*, which causes the material to drop into the trough, from which it is removed by a

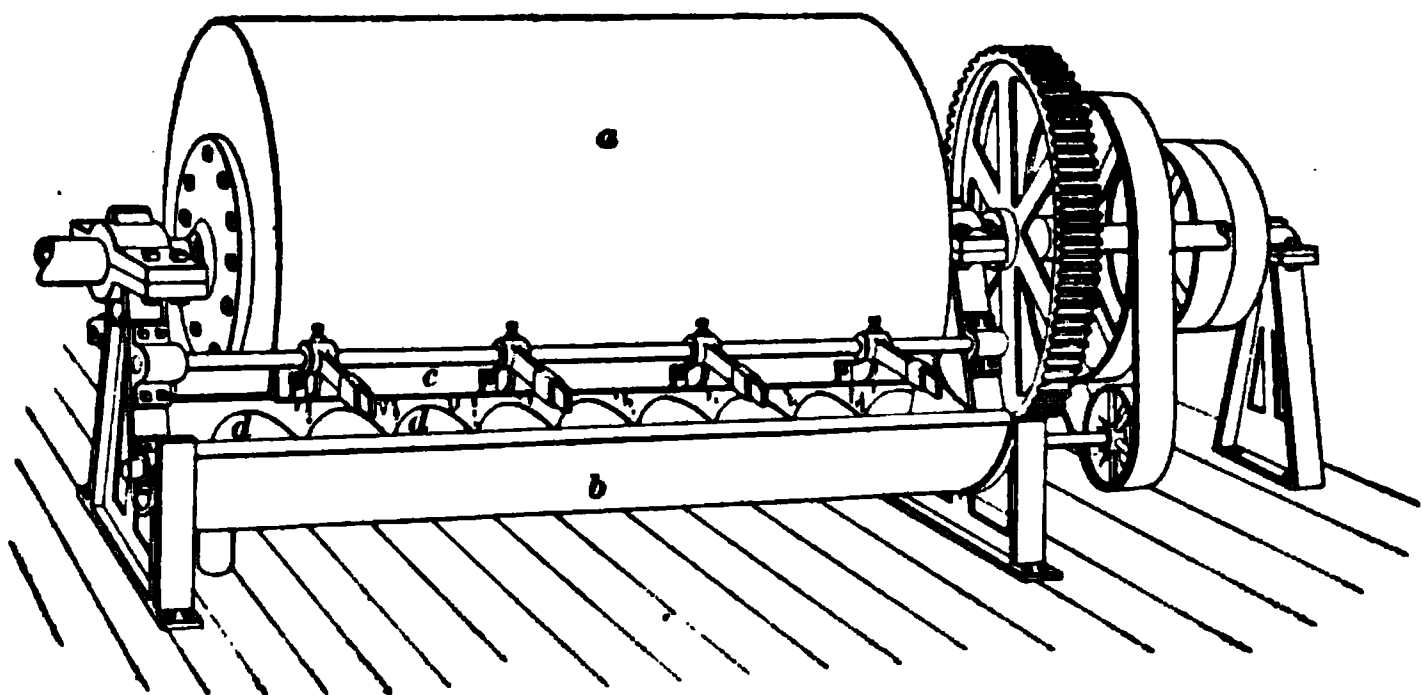


FIG. 1

screw *d* revolving therein to an adjacent agitator, where the lumps formed on the cylinder are broken up. From this agitator it is drawn into the packages. Frequently, the lard compound is removed from the trough by a pump, which draws the material through a screen in the end of the trough, thus breaking the lumps and rendering the stock suitable for immediate drawing into packages. The lard roll is always in connection with the refrigerating system of the plant, to insure a constant cold temperature.

6. The main object in making lard compounds is to have them prepared of as large a percentage of cottonseed oil as possible and yet fulfil the required conditions as to the stiffness of the material to withstand warm temperatures without much softening. Of late years, articles known as "cotto" compounds, such as cottolene, cotto suet, etc., have

been put upon the market as competitors of lard compound. These compounds are practically lard compounds, being made with the yellow, unbleached cottonseed oil instead of the white oil. The proportions and methods of making are identical with the usual compounds, frequently having the oleo stearin substituted by beef fat or suet. In the case of "cotto" compounds in general, the process of bleaching the various materials is not resorted to, the materials being simply mixed and filtered in the manner described.

BUTTERINE, OR OLEOMARGARINE

7. One great outlet for cottonseed oil is its use in the manufacture of oleomargarine or, as it is commonly known, butterine. The quality of oil used for this purpose is that known as butter oil. Oleomargarine was invented in 1870 by Mège Mouries, a celebrated French chemist, on an order from the French government to provide for the army a cheap and wholesome substitute for butter. His discoveries and theories in this line have been accepted and confirmed by the food experts of the world as eminently satisfactory and of great value. He demonstrated by many careful experiments that the formation of butter contained in milk was due to the absorption of fat contained in the animal tissues and that the oil pressed from the sweet fat of beeves corresponded exactly with the oil found in the natural butter. This oil expressed from beef fat is known as *oleo oil* and possesses a bland taste and faint buttery odor. Its color is usually similar to that of butter. While the original name, oleomargarine, was applied only to the product oleo oil, it is now applied to the modern mixture of oleo oil, cottonseed oil, neutral lard, cream or milk, with the usual salt and coloring matter known as butterine. As indicated above, these two names are used in this country synonymously.

The ingredients constituting butterine vary in proportion with the individual ideas of the manufacturers and according

to the season of the year or the climate to which it is to be shipped.

8. Manufacture of Oleo Oil and Neutral Lard. These products are frequently manufactured in connection with the making of butterine. Oleo oil is made as follows: The choicest parts of the beef fat—the caul, ruffle, and heart fat—are chilled and thoroughly washed immediately upon removal from the slaughtered bullock. The fat is chilled for 10 or 12 hours in water, the temperature of which is slightly above freezing, and then removed from the water and allowed to drain for about 1 hour. The material is then cut, or “hashed,” very fine, falling into a water-jacketed kettle provided with mechanical stirring paddles, where it is melted at a temperature not exceeding 170° F. for about 2 hours. The water and scrap settle to the bottom of the kettle, the clear oil on top being siphoned off to another kettle, where it is allowed to settle for 3 or 4 hours, or for even a shorter period. The warm material is then drawn off into receptacles and kept at a temperature of about 90° F. to “grain,” or cause the separation of the solid part of the fat, the stearin, from the liquid portion, the olein, the latter constituting principally the oleo oil. The grained material is after 24 hours “seeding” placed in cloths of heavy, closely-woven duck and subjected to pressing by a powerful press. The press used for this is shown by Fig. 3, *Cottonseed Oil and Products*, Part 2. The solid portion of the fat remains in the cloths and is known as oleo stearin. The oily part expressed is oleo oil, which after a short period of settling is ready to be used for mixing with the other ingredients.

9. Neutral lard is made from the leaf lard of the hog. After chilling in a cold atmosphere for 24 hours it is hashed and cooked similarly to the fat for oleo oil and is separated after cooking in the same manner. The liquid neutral lard is settled free of impurities for about 4 hours, when it is suitable for use in butterine. The temperature used for cooking this material does not exceed 135° or 140° F. Both

oleo oil and neutral lard may be purchased from the large packing houses and prepared for use by simple melting.

10. The proportions of the different ingredients in butterine vary according to circumstances. A large manufacturer of oleomargarine uses the following formula: neutral lard, 35 per cent.; oleo oil, 35 per cent.; cottonseed oil, 15 per cent.; cream or milk, sufficient to produce 15 per cent. of butter. This may be taken as a fairly representative mixture. The highest grades of butterine have from 25 to 33 per cent. of creamery butter in them. Some manufacturers claim to produce butterine without the use of cottonseed oil, substituting for it sesame oil, which answers the same purposes.

11. Process of Manufacture.—The process of manufacturing oleomargarine is carried out on almost identical lines in all factories. The details of the methods are as follows:

The cream, or in most cases milk, is received in felt-covered cans, to guard against fluctuating temperatures. It is at once emptied into a tin-lined vat and when thoroughly mixed is pumped at a temperature of 80° F. into a centrifugal separating machine, which separates the cream from the milk. The latter is now known as skim milk and is of no further use to the butterine maker. The cream is conducted through block-tin troughs to the sterilizer, where it is pasteurized at 170° F. for a few hours to render inert any germs that may have been in the received milk. The cream is then removed and ripened in vats or other receptacles. The period for this is from 20 to 30 hours, the cream being kept at a temperature of 70° F. In warm weather, somewhat less time is required for ripening, or "souring," the cream—the condition necessary for successful churning.

The oleo oil, neutral lard, and cottonseed oil are kept in separate storage tanks adjacent to and generally situated above the churn. The oleo oil is run into the churn in the desired weight at a temperature of 130° F.; the neutral lard is then added at the same temperature, while the cottonseed

oil is added cold or at the room temperature. The ripened cream is taken from the vats at its temperature, 70° F., and added to the other materials, in their respective proportions. The object of adding the ingredients in the churn at these different temperatures is to have the mixture at a temperature of 90° to 100° F. at the time of churning. This point varies with different makers, some preferring a lower temperature, about 80° F., for the churning. Too high a temperature renders this process very difficult, as the butter or oleomargarine will not blend, but will remain separated and granular. In any event, the mixture must be churned under 122° F.

12. The churn used for this purpose is an upright galvanized-iron cylinder—with an open top and having the edges rounded over—about 3½ feet high and 2 feet in diameter, and holds a charge of about 600 pounds. It is provided with paddles attached to a revolving shaft, which is actuated by pulleys and shafting and revolves in a foot at the bottom of the churn.

13. The desired proportions of the different ingredients having been weighed into the churn, the required amount of “butter color” to produce the depth of yellow color desired is added, the paddles started revolving at a very high rate of speed, and the churning continued for about 7 to 10 minutes, when the operation is finished and the butterine formed.

The whole mass is then discharged from the bottom of the churn into vats of ice-cold water, kept at about 33° to 35° F. by means of cakes of ice placed in the bottom of these troughs, or vats. The butterine is allowed to remain here until it sets hard, which requires about 10 minutes, being moved about occasionally by wooden shovels. The chilled mass is then thrown upon inclined tables with the wooden shovels and left there to drain for about 10 hours. The general custom is to leave the material in this condition over night, so that it may be worked the next morning; most factories for this reason do their churning in the afternoon.

The stock while on the draining tables is salted with the usual amount of fine salt, about 8 to 10 per cent. of salt ordinarily being used. A peculiar feature in this regard is the unanimous verdict of oleomargarine makers that Ashton salt, an English production, is the best salt for this purpose. It is universally used by these manufacturers for salting butterine. During the period of draining, the material is turned over several times, to eliminate as much extraneous water as possible. The temperature of the draining room is kept as nearly 70° F. as practicable.

14. The butterine is next taken to the butter worker—a machine for mixing and amalgamating the product to a homogeneous texture and condition. This machine evenly distributes the salt throughout the material, at the same time working out the surplus water. The usual process of making into the desired packages then follows, these varying in size from 1 pound to the usual butter tub or firkin. For export trade the butterine is placed in various sized cans and hermetically sealed. The packages are kept in cold storage until wanted for shipment.

Every oleomargarine factory is under strict government supervision and every package bears a penalty label, similar to other industries, subject to internal-revenue regulations. At the present time ⁽¹⁹⁰²⁾ a tax of 2 cents per pound is placed on all oleomargarine produced. A daily record is kept of all materials used and of all goods made and a monthly report made, under affidavit by the manufacturers, to the United States Internal Revenue Department.

15. The butter color, or coloring principle, used is the same as that used by creameries for genuine butter. It is either annatto (see *Organic Chemistry*) or, more commonly, an aniline color dissolved in cottonseed oil. The proportion used for ordinary trade is from $\frac{1}{2}$ to 1 ounce to each 100 pounds of material, but this varies to suit individual preferences regarding the shade and depth of color suitable.

16. Cleanliness is one of the requisites of this manufacture, as no high-grade butterine can be produced without it. The utensils and all parts of the factory are daily cleaned with hot water and everything used in the whole process is kept always scrupulously clean. In this respect the oleomargarine factory rivals and almost always surpasses the very best creameries in the country.

17. Cultures in Oleomargarine.—Closely associated with the application of bacteriology to butter-making is its application to the preparation of artificial butter and various oleomargarine products. In certain European countries, especially in Holland, oleomargarine is made in very large quantities. The largest factories in the world are located in Rotterdam. In these factories the use of pure cultures has for some time been adopted with almost absolute uniformity. The shrewd business men who manage these factories have thoroughly learned that if they wish to obtain in their products a flavor imitating that of butter, they are obliged to use bacteria to give them this flavor. They therefore buy the artificial pure cultures and inoculate them into large quantities of pasteurized milk in essentially the same way that the butter maker inoculates them in his cream. They allow this milk to stand in a warm place for a length of time, which will produce the proper amount of souring, and then this ripened milk is mixed with the fats and the mixed oils made into oleomargarine products. The result is that a flavor of butter, derived from bacteriological products of the souring milk, is imparted to the oleomargarine. All of the better grades of artificial butter are made in this way. The oleomargarine factories use various kinds of pure cultures and experiment upon them with a good deal more care and know much more about their use than do the butter makers.

Butter makers make comparatively small quantities of butter, while oleomargarine factories make their product in very large quantities, and their market is much more dependent on the grade of their product than is the market

for butter. Some of these oleomargarine factories have their own bacteriological laboratories, where experiments are going on constantly and where they can obtain their own pure cultures and make use of the very best results of the most recent advances in bacteriology.

COTTONSEED FEEDING STUFFS

18. General Remarks.—Cottonseed feeding stuffs include, ordinarily, *cottonseed*, *cottonseed cake* or *meal*, and *cottonseed hulls*. Cottonseed meal has met with great favor abroad for many years, but only of late years has it been consumed to any extent in this country. It is the most concentrated, nutritious, and one of the cheapest of ordinary feeds for cattle and sheep. In the Northern States wheat bran is the bountiful supply of highly protein feed, while cottonseed meal takes its place in the South. Wheat bran contains about 15 per cent. of protein and cottonseed meal about 43 per cent., or nearly three times as much as bran. It is thus an abundant and cheap supply of the protein feed. It is highly esteemed as a feed for all animals except hogs.

Many experiments have shown that cottonseed meal produces, pound for pound, more beef than cottonseed itself, but only a limited number of these experiments are of such a kind as to give anything like a definite idea of the relative beef-producing powers of the two materials. An average of three experiments made showed that 1 pound of cottonseed meal has made as much beef as 1.47 pounds of whole cottonseed.

19. Cottonseed Meal.—Numerous experiments have shown that cottonseed meal produces in the same rations more and cheaper beef than the same amount of corn meal, and generally more than wheat bran, linseed meal, and the other concentrated feeds. The evidence is abundant and the experiments numerous showing that cottonseed meal stands at the head of concentrated feeds. Table I shows the comparative composition of average cottonseed meal and bran.

TABLE I

1 Ton	Water. Pounds	Ash. Pounds	Indi- gestible Protein. Pounds	Carbo- hydrates. Pounds	Fat or Ether Extract. Pounds	Diges- tible Protein. Pounds
Cottonseed meal...	164	144	106	584	262	740
Bran.....	238	116	58	1,258	80	250

One ton of cottonseed furnishes about 750 pounds of cottonseed meal. An analysis of cottonseed meal for feeding purposes gave moisture, 8.72 per cent.; oil, 12.91 per cent.; protein, 43.46 per cent.; carbohydrates, 18.59 per cent.; fiber, 9.72 per cent.; and ash, 6.60 per cent. The composition of cottonseed meal naturally depends on the composition of the seed and the completeness of the separation of the hulls and kernels, and the expression of the oil from the latter, all of which greatly influence the result obtained by analysis.

20. Cottonseed Hulls.—The hulls, constituting about 45 per cent. of the weight of the ginned seed, are composed principally of woody fiber and nitrogen-free extract, which, with water, constitute more than 90 per cent. of the entire substance. The hulls are sometimes ground very fine and bolted, resembling in this condition linseed meal. This finely ground material is at times mixed in varying proportions with cottonseed meal, such as one part of meal to four parts of the hulls, the mixture being sold as cottonseed-meal bran, for a combination food. To make a product resembling English meal as ground from undecorticated seed, which practically is 55 per cent. of meal and 45 per cent. of hulls, the ground hulls have been mixed in the above proportions with the meal and sold as an English product.

The large amount of fiber on the hulls has been claimed to be no detriment as a feed, as it does not, like cattle hair, produce balls in the stomachs of the animals. But the less

lint on the hulls, the better is the product as a stock feed. Cottonseed hulls from different mills, and even from the same mill, vary widely in composition, dependent on the thoroughness of separation from the meats, larger or smaller quantities of the finely broken kernels almost always remaining with the hulls. An analysis of the hulls themselves, free of fiber and meats, gives as an average analysis moisture, 11.36 per cent.; ash, 2.73 per cent.; protein, 4.18 per cent.; fiber, 45.32 per cent.; nitrogen-free extract, 34.19 per cent.; and fat, 2.22 per cent.

21. Cottonseed.—Raw cottonseed has practically never been fed to animals on a large scale. The lint and the dust on the material are apt to prove injurious when fed in this concentrated form, and when fed in excess cottonseed proves deleterious to the health of the animals. An average of twenty-five analyses of cottonseed gave the following result: Moisture, 9.92 per cent.; ash, 4.74 per cent.; protein, 19.38 per cent.; fiber, 22.57 per cent.; nitrogen-free extract, 23.94 per cent.; and fat, 19.45 per cent.

22. Composition of Cattle Feeds.—All cattle feeds, whether concentrated or coarse, like cottonseed meal or hay, are composed of six groups of substances, which the chemist must determine to obtain their value as feed. The term “concentrated feed,” taken in its broadest sense, includes the grains and other seeds of agricultural plants, as well as their many by-products produced in the process of oil extraction and in the preparation of human foods. The groups of substances composing these are *water* or *moisture*, *ash*, *cellulose* or *fiber*, *fat*, *protein*, and *non-nitrogenous* or *nitrogen-free extract*.

Crude ash represents the mineral ingredients of the seed and remains as ashes when the material is burned. This ash consists of lime, potash, soda, magnesia, iron, phosphoric acid, and sulphuric acid; and at times other substances, as chlorine, etc.

Crude fiber, or **cellulose**, is the coarse, or woody, part of the plant or seed. While, as a rule, it is present only to a

limited extent in grains and their by-products, it is largely present in cottonseed hulls.

Crude fat includes not only the various fats and oils found in different feed stuffs, but also waxes, rosins, and coloring matters. It is sometimes termed ether extract, because it represents that portion of the material soluble in ether. The fat found in grains and seeds is comparatively free from these waxes and rosins.

Crude protein is the general name for all of the nitrogenous matters of the seed. It corresponds to the lean meat in the animal. It has the same elementary composition as animal flesh and is considered the most valuable part of concentrated feeds.

Non-nitrogenous or nitrogen-free extract consists of sugars, starch, gums, and the like. The grains are much richer in starch and similar substances than are the cottonseed products.

The fiber and extract matter have the same functions in the process of nutrition, and collectively they are termed *carbohydrates* (see *Organic Chemistry*). The most common and important carbohydrates are sugar and starch. The general methods for the estimation of these substances will be found later.

Some States have statutes requiring that concentrated feed stuffs be sold with a guaranteed analysis as to the percentage of protein and fat they contain. A large manufacturing concern sells its prime cottonseed meal as a feed stuff with the guaranteed analysis of protein not less than 43 per cent.; crude oil and fat not less than 9 or 10 per cent.

Cottonseed meal for domestic trade is always put up in 100-pound bags; that for export either in 100-pound bags or of weight designated by the buyer.

23. Cottonseed as a Feed for Hogs.—Although cottonseed is used with the greatest success and economy in feeding cattle and sheep, it cannot be used as a ration for hogs without deleterious and nearly always fatal results. It has

been used in almost every form for feeding hogs, but never with success when the feeding has been continued any length of time. It has been fed raw, roasted, steamed, and boiled; and the meal, both raw and cooked, has also been tried in various ways, but almost always with the same result—the death of from one-fourth to one-half the drove within 12 weeks from the time the feeding began. Apparently the hogs do well the first few weeks after the feeding begins, but at about the sixth week occasional deaths occur, and the losses continue from that time onwards. The hogs show no indication of sickness and make very satisfactory gains until within 2 or 3 days, or sometimes within a few hours, of death, and no curative treatment seems to have any effect. Nearly every experiment station in the Southern States has endeavored to find some combination of feeds, of which cottonseed should be a part, which could be fed with safety, but without success, and it seems useless to make any further attempt to use it as a hog feed. Before it had any commercial value cottonseed was often thrown into shallow ponds, where it soon became half rotted, and when in that condition hogs often ate it with impunity. With the present market values of the seed, more than double the amount of food can be secured by exchanging it for corn or similar feeds.

Various experiments have been conducted to ascertain what part of the seed—the lint, the hull, or the meal—was injurious to hogs and the manner in which it acted. Recent French investigations show that the injurious effect of cottonseed is due to a poisonous principle peculiar to the kernel.

COTTONSEED FERTILIZER MATERIALS

24. General Remarks.—The principal fertilizing elements in cottonseed products are the well-known combinations of the elements nitrogen, phosphorus, and potassium, which have already been discussed (see *Quantitative Analysis*). These elements are furnished by the cottonseed,

cottonseed meal, and the hulls. The ashes from the latter furnish a large proportion of potash and also phosphoric acid.

25. Whole Cottonseed.—In former years, when cottonseed was practically of no commercial value, the whole cottonseed was used extensively in a wasteful way as a fertilizer in the South, care being taken to first kill the seed by fermentation or by rolling, to prevent germination. At the present time this practice of fertilizing by whole cottonseed has been practically abandoned because of the enhanced value of the seed, and the fact that better fertilizer can be purchased in the form of cottonseed meal and mixed fertilizers. Many cottonseed mills are accustomed to offer to exchange with the producers of the seed a stipulated weight of cottonseed meal for each ton of cottonseed, the quantity of meal offered varying with the ruling prices of seed. This exchange is always advantageous to those using the seed only as fertilizer.

The fertilizing constituents of cottonseed are indicated by the following analysis, which is the average of fifteen analyses of different samples of seed: moisture, 8.42 per cent.; ash, 3.78 per cent.; nitrogen, 3.13 per cent.; phosphoric acid, 1.27 per cent.; and potash, 1.17 per cent. When raw cottonseed is used as fertilizer, the full value of the constituents is not obtained as in the case of meal, as the mechanical condition of a fertilizer exercises a great influence upon its availability. A ton of seed is about equal in value for fertilizing purposes to 800 pounds of cottonseed meal, the superior mechanical condition of the latter being taken into consideration.

The further wastefulness of using cottonseed as fertilizer is evidenced by the fact that when cottonseed is fed to cattle, practically over 90 per cent. of the total fertilizing constituents contained in these as meal or hulls are excreted by the animals, and thus returned to the soil after having filled their function as feed.

26. Cottonseed Meal.—This material is rich in fertilizing constituents and is extensively used as a fertilizer, its mechanical condition being most excellent for application to the soil. Although cottonseed meal contains considerable percentages of phosphoric acid and potash, a large proportion of which is in a state readily available to plants, it is used chiefly as a source of nitrogen in fertilizers. The largest proportion of nitrogen sold in some of the fertilizers in the Southern States is derived from this material, as it is usually a cheap source of the necessary nitrogen.

Cottonseed meal has been applied with excellent results as a fertilizer for sugar cane, cotton, and corn. Its composition with reference to its fertilizing constituents is shown by the following analysis, the average of over two hundred samples being taken: Moisture, 7.60 per cent.; ash, 6.95 per cent.; nitrogen, 7.00 per cent.; phosphoric acid, 2.88 per cent.; and potash, 1.77 per cent. While cottonseed meal, as these facts show, has a high value when applied directly as a fertilizer, the more rational way, as in the case of the raw seed, is to feed the meal to animals, when the large proportions of fertilizing constituents will be recovered, as above noted. An additional benefit also being secured in the production of meat, milk, etc.

27. Cottonseed Hulls.—These hulls, consisting principally of crude fiber and nitrogen-free extract, are hard and dry and are usually covered with a short, woolly lint. They are not frequently used alone as a fertilizer, as the content of nitrogen is low; but they form a good material to place on clayey lands for the purpose of lightening the earth and giving back a large amount of vegetable matter to the soil. An average analysis of the fertilizing constituents of cottonseed hulls is the following: Moisture, 10.17 per cent.; ash, 2.40 per cent.; nitrogen, .69 per cent.; phosphoric acid, .25 per cent.; potash, 1.02 per cent.; soda, .02 per cent.; lime, .18 per cent.; magnesia, .26 per cent.;

sulphuric acid, .08 per cent.; ferric oxide, .03 per cent.; insoluble matter, .05 per cent.

The hulls are usually employed as a fertilizing material in the form of ashes, which vary largely in composition on account of impurities introduced, principally by the use of other fuels in burning them. Most of the potash of cottonseed-hull ashes is in the form of carbonate, a small proportion being present in the insoluble form of silicate, caused by fusion with the sand usually present with the hulls during the incineration. The degree of heat used affects this to a marked degree, from 2 to 5 per cent. being usually present. An average analysis from many samples of hull ashes gave for the principal fertilizing constituents phosphoric acid, 10.85 per cent., and potash, 22.89 per cent. The average moisture for this analysis was 8.95 per cent. The amount of potash is generally between 20 and 30 per cent. The value of cottonseed-hull ashes depends almost exclusively on the amounts of potash and phosphoric acid they contain. This material has come into great demand as a tobacco fertilizer, as the cheap source of the requisite potash is here present and in the form of carbonate is readily available to the plant.

Table II gives the analyses of cottonseed-fertilizer materials given in the preceding paragraphs in a form suitable for comparison.

TABLE II

ANALYSES OF COTTONSEED-FERTILIZER MATERIALS

Material	Moisture	Ash	Nitrogen	Phosphoric Acid	Potash	Soda	Lime	Magnesia	Sulphuric Acid	Ferric Oxide	Insoluble Matter
Cottonseed	8.42	3.78	3.13	1.27	1.17						
Cottonseed meal	7.60	6.95	7.00	2.88	1.77						
Cottonseed hulls	10.17	2.40	.69	.25	1.02	.02	.18	.26	.08	.03	.05
Hull ashes	8.95			10.85	22.89						

METHODS FOR EXTRACTING OIL FROM CAKE

28. Carbon Bisulphide Method.—Although the process of removing completely the oil from the pressed cottonseed cake has not been adopted up to recent times to any considerable extent in this country, the value of the process has been for some time practically demonstrated in Europe as an economical factor in many cases. The process has been used for the extraction of oil in some brands of exported American cottonseed cake with great success. It is very economically applied to cake that is to be used as a fertilizer, as after the extraction of the oil the material is no longer adapted for feed stuff; it is, however, more suitable for fertilizing purposes. The solvent used commercially is carbon bisulphide.

A vertical section of an apparatus designed for this method of extraction is shown in Fig. 2; the boiler for supplying

FIG. 2

the steam required for the distillation of the carbon bisulphide, and for cleaning out the apparatus, working the pumps, shafting, etc. is erected in a separate building, in

order to lessen the risk of igniting the inflammable vapor of the solvent.

A is a lead-lined brickwork chamber into which discharge the tube *f*, leading from the condensers *c*, *c* and the pipe *b*, which will be described later on; this chamber has a capacity of from 8,800 to 11,000 gallons.

The extraction cylinder *B* is of wrought iron and is fitted with a removable cover for charging and emptying the cylinder. It has a capacity of 4,400 gallons, and will take a charge of 12 tons of oil cake. About 8 inches above the bottom it is fitted with a movable perforated false bottom *d*, consisting of wrought-iron plates, the intervening space between the two bottoms being occupied by a perforated steam coil, by means of which steam is blown through the cake after the carbon bisulphide has been drained off.

A second false bottom, composed of a perforated iron disk *d'*, is supported 12 inches from the top of the cylinder by projections from the walls. Both false bottoms are covered with hempen cloth, and the upper one serves to keep the charge of cake in position. An overflow pipe *i* leads from the side walls of the cylinder into the still *C*, into which it delivers the oil and carbon bisulphide from the first-named vessel.

A gooseneck pipe *e*, situated just below the cover of the cylinder, communicates with the condensing coil *c*. Between the false bottom and the actual bottom of the cylinder is a double-branched pipe *h*, communicating with a pump which, through one of the branches, draws up the solvent from the chamber *A* and forces it by way of the other up through the oil cake in *B*. The extractor can be emptied of residual liquid (solvent) by a tapped pipe *b*, leading into the chamber *A*, after the oil cake has been exhausted of oil; while a tube *a* serves to draw off the condensed water in the bottom of the extractor.

The condensing coil *c* receiving the contents of the gooseneck pipes *e* and *e'*, leading from the extractor *B* and the still *C*, is enclosed in a common vessel, through which circulates a current of cold water introduced at the bottom and

escaping through an overflow pipe *g* at the top. The condensed carbon bisulphide falls from the coil *c* through the pipe *f* into the chamber *A*, to be there pumped up again for use as a solvent once more. Evaporation of the bisulphide, and the dangers consequent thereon, must be guarded against by keeping a supernatant stratum of water in the chamber *A*.

The still *C*, capable of holding 1,100 gallons, receives continuously throughout the operation the solution of oil delivered from the extractor by the overflow pipe *i*, and volatilizes the solvent, the oil remaining in the still. The distillation is effected at the outset by a steam coil, direct steam being afterwards admitted to drive off all traces of the carbon bisulphide.

When a sample of the liquid issuing from the extractor shows that all the oil has been extracted from the cake, the solvent remaining in *B* is allowed to run back into the reservoir *A* through the pipe *b*, and after it has all drained away, the portion retained by the cake is driven off by blowing steam in from the jets in the coil under the lower false bottom *d*, so that the vapor of carbon bisulphide is conveyed to the condenser and there liquefied once more, to be collected in the reservoir *A*.

The duration of the extraction is about 12 hours, 2 hours being then allowed for draining; the operation of steaming the cake takes about 8 hours.

The chief cause that has acted against the use of the solvent process for the extraction of oil from vegetable oil cakes has been the difficulty experienced in securing the oil without a characteristic odor; especially so when carbon bisulphide has been used for the extraction. The commercial value of such oils has hitherto been very little, owing to their acid properties and dark green color.

29. Purification of Extracted Oils. — By a recent process such extracted oils are completely freed from all objectionable odor and color, and their value and utility thus greatly increased. The process, which is carried out

with every satisfaction at a good profit, is done as follows :

In a cone-bottomed, cylindrical, upright tank capable of holding 10 to 11 tons of oil is placed about 8 tons of the oil to be treated. The tank is about 13 feet high by 6 feet in diameter, lined throughout with lead $\frac{1}{4}$ inch thick, the joints being soldered together. A cock is placed in the apex of the cone and another in the cylinder, just above the joint of the cone, for drawing-off purposes. Two lead pipes, 2 inches in diameter, descend to the apex of the cone, one for admitting steam, the other for agitating with air, this pipe ending in a funnel-shaped aperture.

Eight tons of oil is charged into the receptacle, and to this is added 600 pounds of chloride of lime, stirred to consistency with twice its weight of water. The whole mixture is vigorously agitated with the blower for several hours; the current of air is then replaced by live steam and 200 pounds of sulphuric acid, diluted with twice its weight of water, is then added. This decomposes the lime soap formed by the first operation. When the temperature rises to 100° C., which requires about 5 hours, the steam is shut off and the mixture allowed to rest. The sulphate of lime and water descend to the bottom of the tank, the separated oil floating over them. The former are withdrawn through the lower cock. The oil, through the treatment, is equal in color to choice yellow cottonseed oil, but contains considerable fatty acids liberated by the decomposition of the lime soap. This material forms an excellent material for soap-making.

30. For other purposes, as illumination, the acidity must be neutralized entirely. This is accomplished in a tank provided with an ordinary stirrer; that described in *Cottonseed Oil and Products*, Part 2, may be used for this purpose. The oil is decanted into this tank and the stirrer set in motion. A weight of 10° Baumé carbonate of soda, equal to 10 per cent. of that of the oil, is now added. After agitating the mixture for about 4 hours, the

neutralization is complete; the soap formed by this operation is precipitated by a 10° Baumé solution of common salt. The mixture is again stirred for a couple of hours, when it is allowed to stand at rest for 24 hours. The mixture separates into three layers, the neutral oil at the top, the soap in the middle, and the water at the bottom. The oil is separated, and is a neutralized oil of good color, ready for sale purposes.

The soap is collected, and when a sufficient quantity has accumulated, the oil is recovered from it by the treatment described for lime soap.

This process is capable of wide application in the treatment of very low-grade cottonseed or other vegetable seed oils. The employment of upright tanks for both these processes is more advantageous in settling than the use of the shallow tanks used in ordinary refining.

RULES REGULATING TRANSACTIONS IN COTTONSEED OIL

REGULATIONS OF NEW YORK PRODUCE EXCHANGE

31. Excerpts from Rules Regulating Transactions in Cottonseed Oil among members of the New York Produce Exchange, adopted April 19, 1900.

RULE 1. *Sec. II.*—Whenever a case is submitted to the Committee (on Oils) involving a tender of more than one hundred (100) barrels, of different shipping marks, the Committee shall test and give its award on each shipping mark separately, unless both parties to the matter in dispute consent to have the different shipping marks treated as one lot. The Committee shall, however, charge only one fee for each case submitted, irrespective of the number of shipping marks involved.

* * * * *

RULE 3. *Sec. I.*—Packages must be good, hardwood, iron-bound barrels, new, or thoroughly cleaned refined-oil barrels, painted or varnished. They must be delivered in good shipping order, and shall not be under 48 or over 58 gallons each, in case of delivery. On delivery of packages other than as above, an allowance not exceeding 50 cents per barrel shall be made by seller.

Sec. II.—A tank car of cottonseed oil shall be considered as not less than 125 barrels nor more than 150 barrels. Differences in weight, either in excess or shortage on tank cars, shall be settled at the market price on the day of shipment, B / L to determine time of shipment.

Sec. III.—A tank car for settlement purposes shall be 125 barrels.

RULE 4.—Tares shall be tested, if required, by either buyer or seller, by emptying 4 barrels of each 100 barrels, to be taken indiscriminately from the lot. Allowance shall be made for difference in tares in excess of 1 pound per barrel.

RULE 5.—Deliveries of cottonseed oil shall be made by weight at the rate of seven and one-half ($7\frac{1}{2}$) pounds net to the gallon, and in lots of not less than 100 barrels at one time and place.

RULE 6.—Deliveries must be made in lots of not less than 100 barrels at any one place, to any vessel, or at any wharf or pier designated by the buyer, provided the vessel, wharf, or pier so designated shall be accessible and within the lighterage limits of the port of New York.

RULE 7. *Sec. I.*—Cottonseed oil must be paid for on delivery of the goods.

Sec. II.—All tenders of oil (other than crude and winter oils) shall be made between the hours of 9 A. M. and 4 P. M., and unless rejected within 24 hours from delivery of sampling order, shall constitute a good delivery.

Sec. III.—All tenders of crude and winter cottonseed oils shall be made between the hours of 9 A. M. and 4 P. M., and unless rejected within 48 hours from delivery of sampling order, shall constitute a good delivery.

Sec. IV.—When oil in barrels is sampled by order of the Committee, the Inspector shall draw samples of not less than 10 per cent. of the lot in question, such samples to be drawn in such a manner as to prevent the introduction of any moisture. A fee of \$2 shall be paid to the Inspector by the party adjudged in fault.

Sec. V.—When oil in tank cars is sampled by order of the Committee, a fee of \$2 for each tank car shall be paid to the Inspector by the party adjudged in fault.

RULE 8.—When a seller fails to notify before 3 P. M., two days before the expiration of the month, of his intention to deliver, it shall be deemed a failure of delivery, and the buyer is privileged to buy to cover the contract at the market price of the day following, holding the seller for any difference.

RULE 9.—Crude cottonseed oil, to pass as prime, must be made from decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer-yellow grade by the usual refining methods, with a normal loss in weight.

RULE 10.—Crude cottonseed oil, to pass as choice, must be made from decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer-yellow grade by the usual refining methods, with a normal loss in weight, and shall test not exceeding 1 per cent. free fatty acid.

RULE 11.—Summer-yellow cottonseed oil, to pass as prime, must be brilliant, free from water and settlings, sweet in flavor and odor, and of straw color, not reddish.

RULE 12.—Winter-yellow cottonseed oil, to pass as prime, must be brilliant, free from water and settlings, sweet in

flavor and odor, of straw color, not reddish, and must stand limpid at a temperature of 32° F. for 5 hours.

RULE 13.—Winter-white cottonseed oil, to pass as prime, must be straw white to white in color, brilliant, sweet in flavor and odor, and must stand limpid at a temperature of 32° F. for 5 hours.

RULE 14. *Sec. I.*—Tests for winter cottonseed oil shall be made as follows: A regular 4-ounce sample bottle shall be filled full of the oil to be tested, a thermometer shall be inserted through the cork of the bottle and hermetically sealed. The oil shall then be heated slowly to a temperature not exceeding 80° F., and remain at that temperature not exceeding 15 minutes. It shall then be chilled until it stands at 32° F., at which point it must stand for 5 hours, and must be clear, brilliant, and limpid at the expiration of that time.

Sec. II.—A fee of \$2.50 for each winter test shall be paid to the Inspector or Tester by the party adjudged in fault.

RULE 15.—Summer-white cottonseed oil, to pass as prime, must be straw white to white in color, brilliant, and sweet in flavor and odor.

RULE 16.—Refined yellow oils, designated as “off,” may be of inferior flavor, and of a color not exceeding orange, or “reddish,” but not “red.”

RULE 17.—All sales of soap stock, unless otherwise specified, shall be on a basis of 50 per cent. fatty acid; provided, however, no soap stock containing less than 45 per cent. of fatty acid shall be a good delivery.

RULE 18.—Settlement of contracts for cottonseed oil shall be made on the basis of 53 gallons to the barrel.

RULE 19.—Settlement of contracts for cottonseed oil shall be made at the mean between the prices bid and asked on the floor of the Exchange on the day of settlement, it being understood, however, that a settlement cannot be

substituted for a performance of contract except by mutual consent.

RULE 20.—Either party to a contract, prior to or upon signing the same, shall have the right to call an original margin of 1 dollar per barrel, and either party may call for additional margins to meet variations in the market of 1 cent per gallon, and all margins called before 12 m. must be deposited before 3 p. m.

RULE 21.—Fictitious sales or false reports of sales are positively forbidden, and will render the parties concerned liable to suspension or expulsion from the Produce Exchange.

RULE 22.—All transactions in cottonseed oil among members of the New York Produce Exchange shall be governed by the above rules, but nothing therein contained shall be construed as interfering in any way with the rights of members to make such special contracts or conditions as they may desire.

RULE 23.—No change shall be made in these Rules by the Committee on Oils before submitting the same to a meeting of the Oil Trade, properly called, at which 8 shall constitute a quorum.

REGULATIONS OF INTERSTATE COTTONSEED CRUSHERS' ASSOCIATION

32. The following rules, governing transactions in cottonseed products, are the basis on which the products of the oil mills, such as oil, cake, meal, etc., are sold. They give, in addition to the rules of the New York Produce Exchange, the requirements of cake and meal, together with the methods of sampling the products and the classification of cottonseed. These rules are very specific and give an excellent grading of the various products. All export and a very large portion of the domestic trade are based on them. They have been adopted by the Interstate Cottonseed Crushers' Association, and also by the Texas Cottonseed Crushers' Association on interstate business.

**RULES FOR THE GOVERNMENT OF TRANSACTIONS IN
COTTONSEED PRODUCTS***Amended and Adopted at Memphis, Tennessee, July 26 and 27, 1899***CRUDE COTTONSEED OIL****MEASUREMENT**

1. A tank (tank car) of cottonseed oil for contract purposes shall be 125 barrels. A barrel of oil if sold loose is 50 gallons. A gallon of oil is $7\frac{1}{2}$ pounds avoirdupois.

2. Crude cottonseed oil may be sold either loose or in barrels, as agreed between the seller and buyer. If in barrels, they shall be good, new, iron-bound barrels properly silitated or thoroughly steamed and cleaned refined petroleum barrels. Packages must be in good shipping order and contain not less than 48 gallons each. On delivery of other than above barrels, an allowance of 50 cents per barrel shall be made by seller.

CLASSIFICATION

3. Crude cottonseed oil shall be classed and graded as follows: Choice crude cottonseed oil must be made from sound decorticated seed, must be sweet in flavor and odor, light in color, free from water and settlings, and test not over 1 per cent. of free fatty acid; shall produce, when properly refined, choice summer-yellow oil, at a loss in weight not exceeding 6 per cent. for Texas oil, and at a normal loss for oil from all other parts of the country.

4. Crude cottonseed oil to pass as prime must be made from sound decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer-yellow grade by the usual refining methods, with a normal loss in weight (provided the oil shall not be rejected for a nominal amount of settlings), but reasonable reduction shall be made in value for all such settlings in excess of $\frac{1}{4}$ of 1 per cent.

5. All oil neither choice nor prime shall be called "off" oil and shall be sold by sample.

REFINED COTTONSEED OIL

6. Refined cottonseed oil shall be classed and graded as follows, summer yellow only being considered.

7. Choice cottonseed oil must be sweet in flavor and odor, of light straw color, clear and brilliant in appearance, free from moisture, and must bleach to a choice white.

8. Prime cottonseed oil must be clear, sweet in flavor and odor, and of yellow color, not reddish, free from water and settlings.

9. Off cottonseed oil includes all oils having any objectionable flavor or odor or a reddish color.

SOAP STOCK

10. All sales of soap stock, unless otherwise agreed upon by buyer and seller, are sold on a basis of 50 per cent. fatty acid, not to fall below 40 per cent. If containing less than 40 per cent. of fatty acid, soap stock shall not be considered merchantable. Delivery to be made in iron-bound, hardwood packages or tank cars.

COTTONSEED CAKE

11. A ton of cottonseed cake is 2,240 pounds, unless otherwise agreed.

12. Cottonseed cake shall be graded and classed as follows: Choice cake must be bright yellow in color, sweet in odor, soft and friable in texture, not burned in cooking, and have no excess of hulls, and must produce when properly ground a bright meal of deep canary color.

13. Prime cake must be of good yellow color—yellow, not brown or reddish—sweet in odor, firm but not flinty in texture, free from excess of hulls, and must produce when properly ground a prime meal.

14. Off cake includes all grades of cottonseed cake which are distinctly off in color, taste, or odor, or which have been improperly manufactured so as to incorporate in it a very large percentage of lint and hulls, or to produce an exceedingly hard, flinty texture.

15. Cottonseed cake, unless otherwise specified, shall be packed in good, strong, sound Dundee bags, either new or second hand, at the option of the seller, unless specified in contract. Packages must be well sewed and in good shipping order.

COTTONSEED MEAL

16. A ton of cottonseed meal is 2,000 pounds unless otherwise stated. A sack of cottonseed meal is 100 pounds gross weight.

Cottonseed meal shall be classed and graded as follows:

17. Choice cottonseed meal must be the product from choice cottonseed cake when finely ground, must be perfectly sound, sweet, and light yellow color (canary), free from excess of lint and hulls. By analysis it must contain at least 8 per cent. of ammonia.

18. Prime cottonseed meal must be made from prime cake, finely ground, of sweet odor, reasonably bright in color, yellow, not brown or reddish, and free from excess of lint or hulls, and by analysis must contain at least 8 per cent. ammonia.

19. Off cottonseed meal includes any cottonseed meal which is distinctly deficient in any of the requirements for prime quality, either in color, odor, texture, or analysis, or all.

* * * * *

COTTONSEED

CLASSIFICATION

Cottonseed shall be divided into two classes: Prime seed and off seed.

21. Prime seed shall be clean, dry, sound seed, free from dirt, trash, and bolls.

22. Seed not coming up to the requirements of prime seed shall be considered off seed. Off or damaged seed shall be settled for on its merits and comparative value as against value of standard prime seed.

GENERAL RULES

23. All offers, sales, or purchases of cottonseed oil (or other cottonseed products) shall be understood, unless specified to the contrary, to be f. o. b. cars at the mill and on the basis of prime quality. Unless specially stated, oil shall be considered as sold loose, and the buyer shall furnish tank cars.

24. All sales of cottonseed products, unless otherwise specified, shall be for cash, payment to be made by resident buyers, on presentation of invoice with railroad ticket signed, or bill of lading attached showing delivery of goods to the carrier, in good order.

25. Payment of non-resident buyers shall be by sight or demand draft, with $\frac{1}{4}$ of 1 per cent. exchange, with bill of lading attached, showing delivery of goods to the carrier in good order, unless otherwise agreed.

26. When goods are delivered to the carrier as agreed, whether in whole or partial completion of trade, payment for same shall become due, if presented during banking hours, and all risks belong to the buyer.

27. On all sales of cottonseed products to or through regular brokers the seller shall pay the brokerage, unless otherwise specially agreed.

28. When a trade is closed with or through a broker, it shall be understood that his fee has been earned, whether the goods are finally delivered or not, provided the failure to deliver arises from the fault of the seller.

29. On all trades by telegraph, day messages requiring day answer shall be open until 12 midnight of the day on which sent. Night messages shall be open till noon following the night on which sent. The time when telegraphs are filed in telegraph office sending same, to govern, and this rule to apply only when no specific time is stated in the original offer.

30. Rules governing trades in cottonseed products are only applicable in the absence of a specific written contract

stating special conditions, but either party to a trade may demand a formal written or printed contract as soon as the trade is completed, such contracts, unless specially excepted, being subject to all the rules of these associations.

31. All trades in cottonseed products shall be either immediate, prompt, or specified dates of delivery.

1. Immediate shall be within 5 working days.
2. Prompt shall be within 10 working days.
3. Specified dates according to contract.

In all cases the bill of lading shall be evidence of date of shipment.

BUYERS' TANKS

32. In case the buyer furnishes tank cars, shipments of same by buyers shall be as follows :

1. Immediate shipment, within 2 days.
2. Prompt shipment, within 5 days.
3. Specified shipment, shall be in such time that, under ordinary course of transportation, cars shall reach seller in time to allow him to make delivery as per contract.

33. If more than one tank car is to be furnished for the same delivery under one transaction, the first car only shall be shipped as above, and the balance shall follow as rapidly as the seller can with certainty load the same.

Seller shall in all cases load cars within 48 hours of arrival at destination.

Seller shall in all cases inspect tank cars and clean them if necessary, at the expense of the buyer, charging only actual cost for same.

TIME CONTRACTS

34. When the time contract is made for any one of the products of a mill, with a date specified for the expiration, and the quantity or quality or both are not stipulated, it is understood to be for all the possible output of the particular products named, that can be made from seed worked up to midnight of the last day named in the contract; the

whole to be put in proper condition for shipment as speedily as possible after the date of expiration of contract.

35. In all time contracts it is understood that the mill is to run to its full capacity and to use every means known to produce goods of the quality stipulated, when so named, and if failure to do either or both of these appears intentional, then both actual and consequential damages may be awarded by the Arbitration Committee.

In case the mill burns, the contract is void.

CLAIMS

36. All claims against shipments of cottonseed products must be made within 5 days after their arrival at American point of destination, except a product for export, in which case 20 days shall be allowed.

37. No claim from any foreign market will be recognized unless the proper samples of the goods are taken and preserved previous to their leaving the American shore, unless samples are drawn before removal from foreign dock, and samples taken according to Rule 36 in claims and rules governing samples.

38. All claims to be brought before the committees of these Associations must be accompanied by an affidavit from a reliable party, substantially in the following form, describing and identifying the samples submitted as taken from and fairly representing the entire shipment.

FORM FOR CLAIMS

39. I, the undersigned, do hereby make affidavit that I have drawn fair and true samples from.....package of, being not less than....per cent. of the entire number of packages embraced in a shipment made by.....fromas evidenced by bill of lading dated.....and issued by.....

The samples were carefully taken so as to secure a fair representation of the contents of the individual package and a true average of the quality of the entire shipment.

I certify to the correctness of the samples which are marked as follows.....and which represent the shipment marked or identified as follows:.....
or contained in..... Sworn to before me,
 a Notary or J. P. of.....County and State of.....
 and duly authorized by law to take depositions, this....
 day of.....189..

.....

SAMPLES

40. Samples representative of any shipment of cottonseed products to secure the official recognition of these Associations or their committees must be secured in substantially the following manner:

41. In the case of oil in tank cars, at least 1 gallon must be taken well down in the body of oil, and from this a 1-quart sample shall be drawn and placed in a perfectly clean tin can, which shall be securely fastened up without the use of sealing wax and carefully labeled, so as to guarantee its identity and correctness.

42. In the case of oil in barrels, a regular 4-ounce sample bottle shall be filled from at least 5 per cent. of the barrels, selected at random, each sample to be from a separate barrel, and so taken as to represent its entire contents. Each sample so taken shall be sealed and labeled as provided above.

43. In the case of cake, sample pieces not less than 3 inches square shall be taken from at least 5 per cent. of the packages in each carload or in the entire lot if not shipped in carload lots, which pieces shall be wrapped in such manner as to keep each lot separate and distinct and fairly representing the shipment from which taken. These samples shall be sealed and labeled so as to thoroughly identify them and the shipment which they represent.

44. In the case of meal, 2 ounces or more from a sack shall constitute a sample of meal and must be drawn so as to fairly represent the entire contents of the bag. Ten or

twenty samples from each carload, or 50 sacks from each 100 tons, if not shipped in car lots, shall be sufficient to represent a shipment. Separate samples of meal should be placed in tinfoil and well wrapped in heavy paper, sealed and labeled so as to identify them and the shipment they represent. Samples of meal, if of approximately same grade and quality, need not be kept separate, but may be commingled, in which case they must be placed in a metal mailing or sample box and carefully marked, showing the number of samples taken, as well as car number and mark.

45. Samples shall in every case be drawn by a reliable party or parties, who shall make affidavit as prescribed by these Rules in the Form for Claims.

46. Samples drawn and presented to the Associations, with all expenses paid in accordance with the above requirements, and with the prescribed form and agreement attached, shall be considered as sufficient evidence for arbitration.

ARBITRATION

The Memphis Merchants Exchange is adopted as the place for arbitrations under these Rules.

ANALYTICAL METHODS APPLICABLE TO COTTONSEED PRODUCTS

INTRODUCTORY

33. In the examination of a cottonseed oil, the first tests invariably applied are of a purely physical and superficial character, consisting of the examination by sight, smell, and taste. Whether or not further supplemented by analytical methods, these are very essential. Although these tests are apparently simple to those familiar with cottonseed oils, to the student desiring to perfect his knowledge and judgment of these products, they are of the greatest importance.

The education and cultivation of these senses should be such that when these tests are applied they will give the student a correct idea of the grade and class of the oil under examination. It is by practice only that this knowledge can be obtained and primarily it must be applied by comparison with standard samples of the different grades of oils. While frequently to the practical oil miller the science of applied chemistry does not appeal, the above tests do and are always applied in his working of cottonseed oil. When supplemented by the usual chemical tests, the grading of cottonseed oils is comparatively easy of acquirement.

The methods of analyzing these products are upon the general lines already pursued by the student. Many of the determinations given can be applied to such products as nitrogen (ammonia), phosphoric acid, potash, etc. in cake and meal. Others, which are especially applicable to certain cottonseed products, will be presented, among them being the methods for ascertaining the amount of crude oil left in the foots, the percentage of oil left in the cottonseed cake after pressing the amount of free fatty acids in the oil, etc. These tests are of the greatest importance in this industry, for it has been by the application of these that great economic changes have taken place in recent years in oil milling and refining. By the application of analytical chemistry, the methods and processes of manufacturing are reduced to a scientific basis—the key to success in this, as in all other similar industries.

The manufacture of cottonseed oil and other products is constantly being improved, largely through the control of the operations exercised by the application of analytical methods.

Cottonseed oil is representative of a class group, occupying a position midway between the drying vegetable oils, as represented by linseed and similar oils, and the non-drying vegetable oils, of which olive oil is a representative. It is classed as a semi-drying oil, acquiring, after long exposure in a thin film, the property of drying. The characteristics of cottonseed oil, as observed in regard to the various

chemical reactions, coloration tests, etc., also occupy an intermediate position between the two groups mentioned. More detailed information regarding this matter will be found in *Quantitative Analysis*.

The following are some of the observed analytical data taken from various sources of cottonseed oil and the fatty acids therefrom:

Crude oil, specific gravity.....	.916 to .920
Refined oil, specific gravity.....	.922 to .926
Refined oil, solidifying point.....	1° C. to 10° C.
Free fatty acids.....	.4 to
Iodine number.....	106 to 112
Maumene's test.....	80 to 90
Saponification value (mmg. <i>KOH</i>) ..	201.6 to 208
Iodine number.....	111 to 115.7
Fatty acids from cottonseed oil:	
Specific gravity at 99° C. (water at 15.5° C. = 1).....	.8467
Specific gravity at 100° C. (water at 100° C. = 1).....	.8816
Titer test.....	32.2° to 37.6°
Melting point.....	35° to 40°

34. Determination of Free Fatty Acids in Oils.
The quantity of lye requisite for the refining of any oil depends on the percentage of free fatty acids present, or as much as desired to extract, all in excess of this being absolute loss. If too much caustic-soda solution is used, in addition to saponifying the free fatty acids, it is likely to cause a saponification of the oil itself and thereby cause a large shrinkage. This is most likely to occur at high temperatures. Further, any excess of lye remaining in the oils treated is lost and wasted, as it must subsequently be removed in the after-treatment.

Commercially, the free fatty acids of vegetable and animal oils are all calculated as oleic acid, although others may be present in small proportions, such, for example, as rosin acids. For technical purposes, such calculation is

sufficiently accurate. The determination is carried out by titrating an alcoholic solution of the free fatty acids with a standard caustic alkali, using phenol phthalein as an indicator. The solutions required are (*a*) a neutralized, dilute alcohol (ethyl or purified methyl), (*b*) an alcoholic solution of phenol phthalein, and (*c*) a standard caustic alkali, either semi-normal or deci-normal. The former is preferable in ordinary working, and in a commercial way is convenient. The alcohol is neutralized by adding to it, drop by drop, a weak caustic solution until it retains after violent shaking the light-pink tint occasioned by neutralization of the acid present in the alcohol. The usual indicator, phenol phthalein, is added before the addition of the caustic. Solutions (*a*) and (*b*) may thus be combined, as is usually done for convenient working. The alcohol is diluted with an equal volume of distilled water.

About 50 grams of oil, or any known weight, is placed in the usual 4-ounce sample bottle and from 60 to 80 cubic centimeters of the neutralized alcohol added to it. The contents are well shaken together, placed on the water bath or in hot water, and heated to about 180° to 190° F. The oil and alcohol are well shaken again to effect the complete solution of the fatty acids. With most cottonseed oils the color will always disappear, as the usual grades have naturally more or less free acid. The semi-normal solution of caustic soda or potash is now added, small portions at a time, until the pink color caused by the caustic disappears very slowly. At this period the solution is added drop by drop until after violent shaking of the sample the pink coloration remains to the alcohol. The end reaction is very sharp and delicate, and the point of neutralization is easily seen in light-colored oils. With crude oils, however, it is necessary to proceed near the finish very cautiously, as the dark color of the oil is apt to mask the end point, causing an excess of caustic to be added. This may be obviated by allowing the contents of the bottle to stand a few moments, when the oil and alcohol will separate, and the color of the latter may then be clearly observed. An example of

titration will make this determination clear. It required for 50 grams of oil 5.5 cubic centimeters of semi-normal alkali to produce the required pink coloration. As 1 cubic centimeter of this solution corresponds to .141 gram of oleic acid, $5.5 \text{ cubic centimeters} \times .141 = .7755$ gram of oleic acid in the 50 grams of oil taken. Hence, $.7755 \div 50 = .0155$, or 1.55 per cent. of free fatty acids in the sample titrated.

35. Test for Mineral Acids in Oil.—Mineral acids are likely to be in cottonseed oils only after refining by an acid process. When the presence of mineral acid is suspected, the oil is agitated with warm water and the aqueous liquid separated. The latter is then tested by adding to it a solution of methyl orange, which will give the usual red or orange coloration if any mineral acid be present.

36. Color Test for Refining Crude Oil.—The solution required for this test is a 20° Baumé caustic-soda solution. Into a large oil-sample bottle 100 cubic centimeters of the crude oil is placed and a few cubic centimeters of the solution added. As this test gives the key to the probable, if not actual, amount of caustic soda to be used in refining, it is performed by adding trial quantities until the desired color in the sample is obtained. To the quantity of oil in the bottle, 2 cubic centimeters of the solution as a trial is added and the whole well shaken together for 1 minute. The bottle and contents are then placed in hot water at 160° F. for 5 minutes. The oil is then filtered into another bottle and compared with a similar bottle of prime yellow oil as a standard. If the color of the filtered oil is not light enough, another test is made in the same manner, using this time 2.25 cubic centimeters of caustic solution. These tests are continued until the standard prime yellow oil is produced. The result is expressed, assuming 2.5 cubic centimeters of solution to have been used to produce the required result, as a color test of 97.5, ($100 - 2.5 = 97.5$).

The practical application of this test may be made apparent by an example. A sample of a tank of 3,000 gallons of crude oil testing 1.5 per cent. in free fatty acids required 3.1 cubic centimeters of the caustic solution to produce a prime yellow color in the sample. The oil has then a color test of 96.9. The quantity of 20° Baumé caustic soda required for producing the standard color in the 3,000 gallons—in other words, to refine it—is ascertained thus:

$$3,000 \text{ gallons} \times 3.1 = 93 \text{ gallons.}$$

This is the exact quantity of caustic soda at 20° Baumé necessary to refine that quantity of oil.

When this strength is necessary to be made weaker, as is often the case, sufficient water is added to make the total caustic solution equal to the desired bulk or percentage of the crude oil, as previously explained.

37. Determination of Free Oil in Cottonseed Foots. About 10 grams of foots are taken and dried free of all moisture (see *Quantitative Analysis*). It is very serviceable to assist this operation by mixing with the foots a quantity of ignited white sand. Where it is desired to ascertain the amount of moisture also, the sand added must be accurately weighed and this weight deducted from the final weight of the dried foots. The thoroughly dried foots, with the sand, is transferred to a filter-paper capsule or thimble and extracted with light-boiling petroleum or rhigolene in the Soxhlet apparatus. The operation is carried out in the manner described later. The solvent is distilled off and the residual oil dried and weighed as there directed. After drying, the foots may also be extracted, as indicated later, and will then give an approximation sufficiently accurate for some technical purposes in the oil refinery or soap factory.

38. Determination of Total Fatty Acids in Cottonseed Foots.—As there is always more or less free oil in the foots, this must first be saponified. Any convenient weight may be taken, care being exercised to first thoroughly mix and amalgamate the oil and soapy matters to a homogeneous

mixture. A convenient weight is 20 grams, as the foots usually contain a large percentage of moisture.

The known weight is transferred to a capacious porcelain or copper dish and about 1 ounce of strong caustic-soda solution placed on it; an addition of 25 or 30 cubic centimeters of methyl alcohol will materially aid in the rapid saponification of the oil while the mixture is being heated. The latter is worked with the spatula while heating in the dish and the materials thoroughly incorporated and saponified at a low heat. This will be accomplished in from 10 to 15 minutes, as a rule. The crumbly soap is stirred with the spatula until the alcohol has all evaporated. The soap is tested for complete saponification by removing a portion and dissolving it in water. No permanent cloudiness or turbidity will be apparent when a thorough saponification has taken place. In the event of incompleteness in this respect, more caustic-soda solution and another addition of methyl alcohol is made to the material in the dish and the whole again heated and stirred. This second procedure, however, is seldom necessary.

When the mass in the dish has cooled, about a liter of hot water is added to it and the whole boiled until all lumps have been dissolved and a clear solution of soap is obtained. To this solution is then cautiously added dilute sulphuric acid in slight excess, when the fatty acids will separate and rise in clots to the top. The boiling of the contents of the dish is continued, with constant stirring to avoid loss by spurting, until a perfectly clear layer of the melted fatty acids appears on the surface.

The underlying acid water may be siphoned off from the fatty acids, care being taken, however, to avoid removing any of the fatty acids in this operation. The latter are washed several times with hot water and the siphoning repeated until the excess of sulphuric acid used has been thoroughly removed. The fatty acids are then transferred to a weighed dish, any adhering material being removed from the dish by light-boiling petroleum, or rhigolene, and added to those already in the weighed dish. The fatty acids are

transferred to a drying oven and dried at 110° C. to a constant weight and the percentage calculated as given below.

Instead of siphoning from the dish, the following method may be applied, which in very accurate operations is to be preferred. After cooling somewhat, the contents of the dish are poured into a separating funnel of sufficient capacity and the underlying water withdrawn through the stop-cock. The dish that contained the fatty acids is now rinsed with the usual solvent and the washings placed in the funnel with the rest of the fatty acids. The washing of the dish is continued until all traces of the fatty acids have been removed. They are now washed in the funnel with hot distilled water until all traces of sulphuric acid have been removed from the fatty acids by the successive washings. The latter should be saved for further treatment. They are now drawn off into a tared beaker or capsule and dried, as usual, at 110° C. to constant weight. The separatory funnel must be rinsed in the same manner as the dish and the rinsings added to the fatty acids in the dish.

The wash water drawn off, if inconveniently of too great bulk, is evaporated to a convenient quantity and shaken up several times with the petroleum ether or rhigolene to remove any fatty acids that may remain in them, which may be equal to $\frac{1}{2}$ per cent. or more of the weight of substance taken. The solvent is withdrawn into either the receptacle containing the bulk of the fatty acids or it may be evaporated in another weighed dish and dried in the usual way. In the latter case, the two residual weights must be added to obtain the total weight of the fatty acids. From this weight the percentage of total fatty acids in the original foots can be readily calculated in the usual way by dividing the weight of substance obtained by the original weight of the sample taken and multiplying by 100 (see *Quantitative Analysis*).

39. Determination of Fatty Acids in Soap Stock. By means of the preceding method the fatty acids of soap stock may also be determined. In place of the preliminary

saponification, the material may be dissolved at once in water and the operations followed as directed above. Cottonseed soap stock to be merchantable must contain at least 40, and in some cases 45, per cent. of fatty acids.

40. Cold Test for Cottonseed Oils.—Winter cottonseed oils are always sold subject to this test and must fulfil the requirements set forth in the Rules of the New York Produce Exchange. The oil to be tested must be clear and have any stearin present completely dissolved in the oil at the time of testing. This test is simply accomplished by allowing the sample of oil to remain for 5 hours at a temperature of 32° F., at the expiration of which period the oil must appear clear, brilliant, and limpid.

It is at times desirable to ascertain the actual cold test of a cottonseed oil for certain specific uses. The method applicable in such cases is the following: A 4-ounce sample bottle is filled about half full of the oil to be tested and a thermometer, passing through a cork, is extended well into the oil. The bottle thus arranged is placed in a freezing mixture and the oil frozen or congealed. The bottle is then withdrawn and, protected from the heat of the hand, the oil is stirred by the thermometer until it runs freely from one end of the bottle to the other. The thermometer is then withdrawn from the bottle and wiped with a piece of cotton waste sufficiently to ascertain the temperature. The reading may be taken as the actual cold test of the oil; in other words, the temperature at which the oil will remain limpid and flow without reference to its clearness.

41. Determination of Mealy Matter in Crude Oil. While merchantable crude oils, as a rule, are free from this material, which in reality is but fine cottonseed meal, some imperfectly settled or filtered oils contain notable amounts over that allowed by rules governing trade transactions. The determination of this is a simple operation.

A known quantity of the crude oil is taken and filtered through a dry and previously weighed filter paper. The operation is materially shortened, as to the time required,

by mixing the sample with twice or three times its volume of carbon bisulphide. The filter paper retains the fine meal; the filter is then washed with successive portions of carbon bisulphide, dropping it around the top edge of the paper until the latter is washed thoroughly free of oil. The filter is dried as usual in the drying oven and then weighed with the adhering meal. The increase in weight is the amount of mealy matter present in the sample of crude oil taken. The percentage is found in the usual way.

Choice crude oil should be entirely free from this material, while one-fourth of 1 per cent. is allowed in prime crude oil. The usual care should be taken in sampling to secure a fair average and representative sample of the crude oil to be subjected to analysis.

42. Determination of Moisture in Cake or Meal. The cake is made into meal in the manner described later. Five grams are taken and dried in the usual manner in the drying oven to a constant weight, which ordinarily requires about 3 hours at 105° C. The loss in weight is regarded as moisture (see *Quantitative Analysis*).

43. Determination of Oil in Cake or Meal. — The average oil mill seldom makes an accurate analysis of the amount of cottonseed oil left in the pressed cake or ground meal. Not that analyses are not made, but they are more or less crude; sufficiently accurate, however, for working purposes in the mill, where strict accuracy is not as important as an immediate knowledge of the effective work the presses are doing. Both the mill-working method of analysis and the method carried out by the analytical chemist will be described, as the student should be familiar with both.

44. The *accurate determination* of oil in cake or meal is carried out as follows: The manner of taking an average sample influences to a remarkable extent the final result; this is especially true in the case of the oil cake, which must be reduced to a fine powder before weighing. The cake selected for the test is sawed through diagonally both ways, and equal portions again sawed from each of these

pieces, care being taken to have as representative a sample as possible from all parts of the cake—the center, middle portion, and the edge. The meal dust and selected pieces may be separated by passing through an 80-mesh sieve. The fragments in the sieve are reduced to fine powder in a porcelain mortar and again screened, and this procedure followed until all the portion of the cake selected for analysis has been passed through the screen. The sample is then thoroughly and intimately mixed as usual with materials of this nature. Five grams of the meal or any definite weight, as 20 grams, are accurately weighed off in a small porcelain dish and dried to a constant weight, as in the determination of moisture, previously given.

The dried meal is then transferred into a folded filter paper or a filter-paper capsule (the latter being the more preferable), and placed in the Soxhlet extraction apparatus. The solvent used may be ether, light-boiling petroleum, or carbon bisulphide. The latter is in most general use for this determination. The meal in the capsule *c* is kept from being carried over with the solvent by a wad of absorbent cotton placed on top of it. The general arrangement may be seen in Fig. 3, in which *b* is a tared flask containing the solvent, *a* the Soxhlet extraction apparatus, *d* the condenser cooled by appropriate water supply, and *e* the filter-paper capsule containing the cottonseed meal from which the oil is to be extracted. The capsule containing the meal having been placed in the tube, the solvent is placed in the flask, the weight of which is accurately known, and the heat applied to boil the solvent. As the solvent boils, the

FIG. 3

vapor is condensed and falls directly into the capsule on the meal, remaining in contact with it until the solvent is siphoned off by the attachment *c* on the apparatus. The solution passes down into the flask to be again boiled and recondensed, this operation being continued until all the oil has been extracted, which usually requires, if the extraction is regular, about 1 hour. By properly arranging the source of the heat, the operation is performed regularly and automatically.

As all these solvents mentioned are very inflammable, great care must be taken, especially when heating by the naked flame, to have the corks of the apparatus air-tight and a perfect condensation of the solvent. As carbon bisulphide has a very low boiling point, in the oil mill it is far preferable to have steam or hot water as the source of the heat, which is amply sufficient to guard against undue risks of fire by possible accidents through breakage.

When the extraction is complete, the Soxhlet apparatus is disconnected from the condenser *d* and the thimble *e* removed. The apparatus is then set up as before and the volatile solvent distilled into the space that the thimble with the contained meal occupied in the process of extracting the oil. When the solvent has nearly reached the level of the top of the tube *c*, the apparatus is again disconnected and the distillate emptied into a convenient bottle. This operation is continued until the greater portion of the solvent used in the extraction of the oil has been recovered. The redistilled material may thus be saved and utilized for further extractions. The residue in the flask is then placed in the drying oven at a temperature of 105° C. to drive off the last traces of the solvent from the extracted oil. From 2 to 3 hours, depending on the quantity taken for analysis, is sufficient for this. The usual calculation, dividing the residual weight by the weight of substance taken and multiplying by 100, gives the percentage of cottonseed oil in the cake or meal. In the analysis of cottonseed meal, as the material is already ground, it is only necessary to have it of

the fineness mentioned and to insure an average representative sample being taken.

45. The *approximate determination* of oil in cake or meal is fairly accurate and adapted for working results in the oil mill and well adapted for rapid results closely approaching the truth.

The sample is prepared in the same manner as has been described. A known weight of meal is placed in tube *a*, Fig. 4, about 10 inches long and $1\frac{1}{2}$ inches wide, the lower part drawn out to a tube about 2 inches long and $\frac{1}{4}$ inch in diameter. The material *b* is packed fairly tight upon a wad of absorbent cotton *c* in the bottom of the large tube and another wad of cotton placed on top of the material. The latter plug is serviceable for removing any fine particles of meal remaining on the weighing glass. A weighed porcelain dish having been placed underneath the tube, the latter supported in an upright position, is filled with carbon bisulphide. The solvent slowly percolating through the meal dissolves out the oil readily, the solution dropping into the tared capsule beneath. After the first addition of the solvent has gone through the meal, successive additions in the tube are made until the carbon bisulphide comes through colorless, when all the oil has been extracted. As the solvent evaporates very rapidly, a small-sized dish is sufficient to contain the solution of oil.

When all the oil has been dissolved out, the end of the tube is washed with a small quantity of the solvent to remove any adhering oil. The solution of oil in the dish has the carbon bisulphide driven off and then dried in the oven at 105° C. to a constant weight. The usual calculation gives the percentage of oil that was in the cake or meal. During

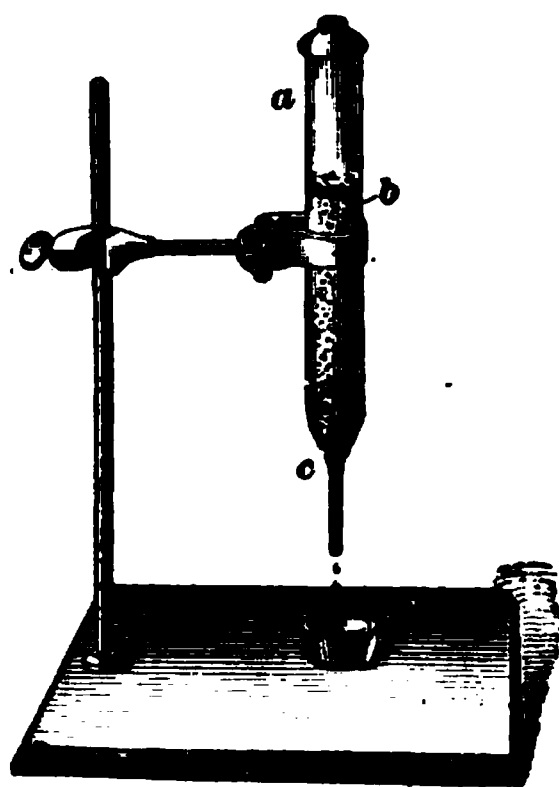


FIG. 4

the operation the tube is kept covered with a suitable porcelain crucible cover to prevent a rapid evaporation of the solvent. Extreme caution must be taken to have no fire or flame in the vicinity where this operation is being carried out.

The dimensions of the tube may be varied to suit individual convenience; in some mills a tube 15 inches long and $\frac{1}{8}$ inch wide is used.

46. Determination of Fertilizer Constituents.—The usual estimations for nitrogen, phosphoric acid, and potash, described in *Quantitative Analysis*, are applied to cottonseed cake and meal. For the estimation of nitrogen, the Kjeldahl method should be employed, as it is the most convenient (see *Quantitative Analysis*).

The following modification of the Kjeldahl apparatus has been found very serviceable. In place of the usual simple bent tube extending from inside the flask *A* into the condenser *d*, the bulb *a*, as shown by Fig. 5, is employed. The tube *b* is blown in the bulb *a*, its shape allowing of rapid and violent boiling without incurring the risk of having any particles of the alkaline solution projected and carried over into the distillate of ammonia. The bulb is drawn out to a tube end *c*, which passes through a cork into the flask *A*.

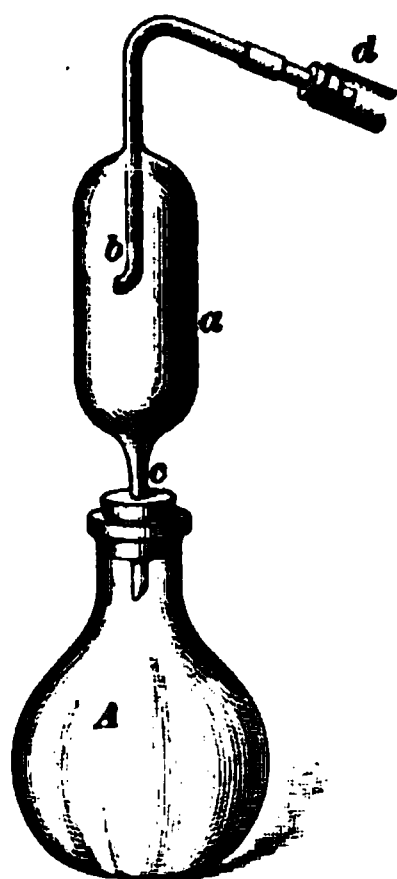


FIG. 5

wise deteriorated. See also Table II.

An average analysis from many samples of cottonseed meal, made from decorticated seed, showed the following percentages of fertilizer constituents: nitrogen, 7.25 per cent. (equivalent to ammonia 8.80 per cent.); phosphoric acid, 2.24 per cent.; potash, 1.74 per cent. These percentages will vary, naturally, with the amount of moisture present in the meal, which usually is about 6 to 7 per cent., and with the quality of the seed originally employed, whether sound, heated, or otherwise

ANALYSIS OF MEAL OR CAKE FOR FEED-STUFF CONSTITUENTS

47. Determination of Protein.—The nitrogen in the substance is determined by the Kjeldahl method in the usual way and the percentage of nitrogen multiplied by 6.25, the factor employed for this purpose. The nitrogen may be determined by any desired method, but this method has been found most convenient.

48. Estimation of Crude Fiber.—Two grams of the substance are extracted with ordinary ether, or the residue from the ether-extract determination may be used for this. To this residue in a 500-cubic-centimeter flask, add 200 cubic centimeters of boiling sulphuric acid of 1.25 per cent. ; connect the flask with an inverted condenser, the tube of which passes only a short distance beyond the rubber stopper into the flask. Boil at once and continue the boiling for 30 minutes. A blast of air conducted into the flask serves to reduce the frothing of the liquid therein. After the required period of boiling, the liquid is filtered and washed with boiling water until the washings are no longer acid. The substance on the filter is rinsed back into the same flask with 200 cubic centimeters of a boiling 1.25-per-cent. solution of caustic soda, free, or nearly so, from carbonate of soda. This solution is at once boiled again for $\frac{1}{2}$ hour in the same manner as for the treatment with acid. Filter on a Gooch crucible and wash the substance until the washings are neutral. The crucible and contents are dried at 110° C. and the weight noted. The substance on the crucible is completely incinerated and the crucible cooled and again weighed. The loss in weight represents the crude fiber in the material.

The filter used for the first filtration may be linen or any other form that secures clear and reasonably rapid filtration. The solutions of sulphuric acid and caustic soda are to be made up of the specified strength, determined accurately by titration and not merely from specific gravity.

49. Determination of Ash.—Char from 2 to 3 grams of the substance in a platinum crucible and burn to whiteness at the lowest possible red heat. For this purpose the ordinary muffle furnace is very convenient. If a white ash cannot be obtained in this manner, exhaust the charred mass with water and collect the insoluble residue on a filter. This is burned and this ash added to the residue from the evaporation of the aqueous extract. The whole is then heated to a low redness until the ash is white or nearly so.

50. Determination of Ether Extract.—To prepare the anhydrous alcohol-free ether required for the estimation of fat, any of the commercial brands of ether are washed with two or three successive portions of distilled water and solid sticks of caustic potash or soda added until most of the water has been extracted from the ether. Carefully cleaned metallic sodium is cut into small pieces and added until there is no further evolution of hydrogen gas. The ether thus dehydrated must be kept over metallic sodium, and should be only lightly stoppered in order to allow any accumulating hydrogen gas to escape. The ether may be drawn off with a pipette as required.

(1) *Determination by Direct Method.*—From 2 to 3 grams of the substance, after drying, are extracted with anhydrous alcohol-free ether for 16 hours. The extract thus obtained is dried as usual to a constant weight. This is termed the ether extract, which is sometimes designated as crude fat.

(2) *Determination by Indirect Method.*—The moisture is determined as described in the following article and the dried substance extracted in the Soxhlet apparatus for 16 hours. The substance is again dried and the loss of weight regarded as ether extract, or crude fat.

51. Determination of Moisture in Feeds.—The substance is ground and passed through a sieve with circular holes 1 millimeter in diameter. The sample prepared in this manner is used in all the determinations for feeding stuffs. From 2 to 3 grams of the material is dried for 5 hours, at a temperature of boiling water, in a current of

hydrogen or in vacuo. If the substance is contained in a glass vessel, the latter must be kept from contact with the boiling water. The loss in weight will be the moisture contained in the substance.

52. Determination of Carbohydrates. — These are determined as (a) the reducing sugars (estimated as dextrose), (b) the sucrose, and (c) the starch (see *Quantitative Analysis*, where the methods for the determination of sugars are given in full). In the analysis of cattle feeds, 3 grams of the prepared sample is stirred in a beaker with 50 cubic centimeters of water for 1 hour. It is then filtered into a 250-cubic-centimeter flask, washed, and the solution made up to the mark.

(a) The *sugar* is determined as dextrose, according to the methods described in *Quantitative Analysis*. If the solution be difficult to filter clear, 2 cubic centimeters of alumina cream should be added.

(b) The *sucrose* is determined in 50 cubic centimeters of the filtrate obtained above in the manner described in *Quantitative Analysis*. Before calculating the invert sugar obtained as sucrose, the reducing sugar obtained as dextrose must be deducted.

(c) The *starch* is determined in the insoluble residue from the filtration of the sample of 3 grams taken. The residue is heated for $2\frac{1}{2}$ hours with 200 cubic centimeters of water and 20 cubic centimeters of hydrochloric acid (1.125 sp. gr.) in a flask provided with a reflux condenser. The contents of the flask are then cooled and neutralized with sodium carbonate. The volume of solution is made up to 250 cubic centimeters, filtered, and the dextrose determined in an aliquot portion of the filtrate. The weight of dextrose obtained multiplied by .9 gives the weight of the starch.

In this method there will be included as starch the pentoses and other carbohydrate bodies present, which suffer hydrolysis and conversion into reducing sugars on boiling with hydrochloric acid.

(d) Starch may also be estimated by the following method, which has recently been devised; it gives good results in the determination of this carbohydrate. According to L. Gianturco, the aluminum hydrate thrown down from an aluminum-salt solution and dried at 100° C. corresponds with the formula $Al(OH)_3$. When calcined in a platinum crucible, it changes into Al_2O_3 . If starch is suspended in water, and to this mixture is added a definite quantity of a standardized solution of an aluminum salt and the aluminum is precipitated as hydrate by means of ammonia, the starch is carried along by the aluminum hydrate, the precipitate being gathered on a filter and washed with as little water as possible until the wash water contains no more sulphates. If this mixture is dried and weighed, the difference between the weight found and the known weight of the aluminum hydrate, corresponding to the used volume of alum solution, furnishes the weight of the starch. Finally the dried deposit may be calcined in a platinum crucible to a constant weight, whereby the aluminum oxide Al_2O_3 + ash of the starch remains as residue. If by this method a considerably larger amount of aluminum oxide should be found than can be computed from the employed amount of the titrated alum solution, the presence of a considerable quantity of insoluble mineral substances would be proved in the starch. The alum solution employed by L. Gianturco contained .060769 gram of the salt per cubic centimeter, corresponding with .01 gram of aluminum hydrate. It is claimed that this method gives better results than any other estimation method.

53. Rapid Method of Estimating the Iodine Number in Oils, Greases, etc.—The following recently devised modified method, originally carried out with greases and lard, has been found very serviceable and reliable for oils. For a quick determination of the iodine number in greases, J. Ballier uses a solution of 50 grams of iodine and 32 grams of bromine in glacial acetic acid, or a solution of 33.5 grams of iodine and 42.2 grams of bromine in the same solvent, saturating the liquid, which amounts to 950 cubic

centimeters, with mercury bichloride (about 95 grams being required). The solution is ready for use in 2 days, and is so made by diluting with glacial acetic acid that 1 liter of it corresponds to 100 grams of iodine; i. e., that 5 cubic centimeters of the bromine-iodine solution requires 39.4 cubic centimeters of $\frac{n}{10}$ thiosulphate solution for discoloration.

As, a solvent for the greases, a mixture of 500 cubic centimeters each of glacial acetic acid and chloroform is used. This is saturated with mercury bichloride, and 10 cubic centimeters of a 10-per-cent. solution of potassium iodide and enough bromine-iodine solution as is required for the liquid to remain colored a yellowish red, are added.

In making the determination, dissolve 1 gram of grease or oil in 20 cubic centimeters of the solvent mixture until the resulting yellowish-red tint remains constant for at least 5 minutes. The results are said to coincide approximately with those of the Von Hübl method. The computation is as follows: Since 1 gram of fat or oil is used and the bromine-iodine solution contains exactly 10 per cent. of iodine, or an equivalent quantity of bromine, the iodine value is found by multiplying the number of cubic centimeters of the bromine-iodine solution used by 10. In order that the standard of the bromine-iodine solution remains constant, it is well to charge the burette by means of a pipette, and not by direct pouring in.

For the usual method of determining the iodine number, the student is referred to *Quantitative Analysis*.

54. Detection of Cottonseed Oil in Mixtures.—Among the most recent contributions to the scientific investigation of cottonseed oil is that of Raikow, who has demonstrated that this oil contains organic chlorated compounds. Upon this characteristic, which is not present in olive oil or nut oils, a new method for the identification of cottonseed oil in mixtures is based. This is especially applicable to mixtures of olive oil and cottonseed oil.

(a) The *presence of chlorine* in cottonseed oil may be detected by burning it and collecting the products of combustion in a solution of caustic potash free from all traces of chlorine and sulphur. The solution is then acidulated with pure nitric acid, and precipitation of the chlorine present effected with nitrate of silver.

(b) The *presence of sulphates* may be detected by boiling the solution obtained with the combustion products, and after acidulating it with hydrochloric acid adding the solution of barium chloride in the usual manner. Although it has been claimed by some that cottonseed oil contains volatile sulphureted compounds, the most recent investigations on oils from many different localities have failed to confirm their presence. The organic chlorated compounds present in all cottonseed oils are not soluble in water and only slightly soluble in alcohol.

Where the mixture of cottonseed oil with olive or nut oils is small in quantity, a larger amount of the mixed oil must be taken for analysis. In such case, about 4 or 5 grams are burned and the products of combustion collected by aspiration and passage through a wash bottle containing potash solution (see *Quantitative Analysis* for the adaptation of the apparatus necessary for this procedure). In the average mixture of these oils, 1 gram is sufficient for analysis. The entire absence of chlorine compounds in olive and nut oils and the presence of these compounds in cottonseed oil gives to this test a positiveness that has heretofore been lacking in the various methods of examining mixtures of olive and cottonseed oils, where the latter is present only in small quantities.

The quantity of chlorine in different cottonseed oils varies largely, and up to the present time it has not been definitely ascertained whether these compounds preexist in the oil or are formed at the time of its preparation.

MANUFACTURE OF LEATHER

INTRODUCTORY

1. Leather.—By the term **leather** is understood the product obtained by the treatment of hides or skins of animals with certain reagents that convert the animal tissues into a tough, opaque, and fibrous substance, which is unaffected by boiling in water and which does not undergo putrefaction. The process by which this is accomplished is known as **tanning**.

The entire substance of the skin is not affected by tanning agents, but only that portion known as the *corium*, or inner skin. The structure of the skin will be fully explained later.

The action of tanning agents is one of chemical or mechanical combination with the fibers of the corium so that on drying they do not agglutinate and form a stiff mass.

2. Tanning Agents.—These substances may be grouped into three divisions, as follows:

1. *Tannins of the Vegetable Kingdom.*—These are organic substances containing tannic acids and are obtained from barks, seed pods, and other portions of certain trees. The ground material, its infusions, and its dried extracts are also used.

2. *Mineral Salts.*—Tanning by means of these substances is known as *tawing*. Alum and sodium chloride are used in some processes, also salts of iron and chromium.

3. *Oils and Fats.*—Various kinds of fish oils and tallow are used in tanning chamois skin and in making the so-called oil-tanned leather.

3. The tanning industry includes a number of subtrades necessary to the production of different kinds of leather, each of whose practices differ widely. They have a common starting point—the raw hide—and this varies from the delicate lambskin for fine gloves to the heavy ox hide for men's shoes. The process for tanning the lambskin into leather would not do for treatment of an ox hide, and so the different divisions of tanning have not all progressed at the same rate, and in any consideration of the subject each branch should be taken up separately.

There are some operations that are followed, with more or less variation, in all kinds of tanning, and an explanation of these operations, preceded by a discussion of the materials used, will make later reference understood.

TANNING MATERIALS

4. *General Remarks.*—With a view to increasing the number of vegetable tannins available for tanning, diligent researches have been going on for some years. As a result, the number of tanning materials has been greatly increased and their cost lessened.

The economical leaching of bark of late years has served to lengthen the time during which our native barks will be available; the use of wood extracts has also served to protect and increase the life of bark tanning, and as the supply of wood from which these extracts are made is practically unlimited, this generation has nothing to fear as to a shortage of tannins.

5. *Vegetable Tanning Materials.*—In the tanning of sole leather, the following vegetable tanning materials are most commonly used:

Hemlock bark is the most important tanning material used in America, about 70 per cent. of all leather made being tanned by it. It is the bark of *Abies Canadensis*, and in the East contains about 11 per cent. of tannin. The Western hemlock may run as high as 17 per cent. of tannin. Leather tanned with it is tougher than that tanned with oak bark, but is not so pliable. Both solid and liquid extracts of hemlock bark are on the market.

Oak bark is also one of the most important tanning materials. It is the inner bark of several varieties of the oak tree, as the English oak (*Quercus Robur*), the rock-chestnut oak (*Quercus monticola*), and the yellow oak (*Quercus tinctoria*). White oak (*Quercus alba*) and the red oak (*Quercus rubra*) are not so valuable, because of their lower tannin content and undesirable color. The tannin of the several varieties of oak is known as quercitannic acid $C_{17}H_6O_6$. There are four anhydrides of this acid, the first, phlobaphene $C_{34}H_{30}O_{17}$, the second $C_{34}H_{28}O_{16}$, the third Oser's oak red $C_{34}H_{26}O_{15}$, and the fourth Löwe's oak red $C_{34}H_{24}O_{14}$. Quercitannic acid and phlobaphene only are of importance in tanning.

Canaigre (*Rumex hymenosepalus*) is a native of the arid Southwest. Some recent attempts have been made to cultivate it and introduce it as a tanning material, but with little success. The coloring matters and starch that are extracted from it affect the quality of the tanned leather.

Quebracho is the name of several hardwood trees growing in South America. The wood and bark of *Quebracho Colorado* contain from 15 to 20 per cent. of a bright-red tannin, but does not contain enough non-tannins to aid the formation of fermentive acids necessary to plumping, nor will it yield a full, well-nourished leather unless combined with other forms of tanning agents richer in non-tannins.

Palmetto root is obtained from the palmetto tree found abundantly in the Southern lowlands. It is attracting considerable attention among the Southern tanneries. The root is cut up and treated like bark. Its action is quick and leather tanned with it is tough.

Gambier (*pale catechu*) is an important tanning agent. It is the dried extract from the leaves of *Uncaria gambier* and *Uncaria acida*. The extract is readily soluble in warm water. It contains from 36 to 40 per cent. of a brown tannin that rapidly penetrates leather and tends to swell it; taken alone, it produces a soft, porous tannage. It is largely used with other materials for tanning both light and heavy leathers. It is exported from Singapore in pressed blocks and cubes.

Catechu, or *cutch*, is the dried extract from an East Indian tree, *acacia catechu*. It contains from 45 to 55 per cent. of a special variety of tannic acid. The extract is evaporated until a thick, dark-brown product is obtained, which solidifies on cooling. In this form it is exported.

Gallnuts, or *nutgalls*, are excrescences on certain plants, caused by gall flies, which puncture the bark in order to deposit their eggs. Oak gall, or Aleppo galls, and Chinese galls are the most important. The oak gall is formed by the insect on *Quercus infectoria*. The eggs hatch and the larva develops within the gall. The best galls are gathered before the fly becomes fully developed, and contain from 60 to 70 per cent. of gallotannic acid. The Aleppo galls are the most valuable.

Sumac in all its varieties is a common and valuable tanning agent. It consists of the powdered leaves, stems, and other parts of the so-called tanners' sumac (*Rhus coriaria*, *Rhus typhina*, and other varieties of *Rhus*). The best sumac comes from Sicily. Italian, Spanish, and French sumac is found on the market. It contains from 12 to 25 per cent. of tannin. The American variety, although containing from 6 to 8 per cent. more tannin than the European, is not so valuable on account of a dark coloring matter contained in it, which injures light leathers. When used it is generally mixed with the imported sumac.

Valonia is the commercial name for the acorn cups of several species of oak (*Quercus ægilops* and *Quercus macrolepis*) coming from Asia Minor and Greece. They contain from 25 to 35 per cent. of tannin, somewhat resembling that of

oak bark, but giving a browner color and heavier bloom. It makes a hard, water-resisting leather and is sometimes mixed with oak bark for sole-leather tanning.

Divi divi is the dried seed pods of a South American bush. The pods are about 3 inches long, of a brownish color, and bend in the form of the letter S in drying. These pods contain from 30 to 50 per cent. of a peculiar tannin, somewhat similar to that of valonia.

Myrobalans are the dried fruit of several species of *Terminalia* from Hindustan, Ceylon, etc. It contains quite a large percentage of tannin. It is generally used mixed with oak bark to modify its color and produces a soft tannage.

6. Classification of Tannins.—The tannins in tanning materials are different members of a large group of organic bodies, known chemically as *tannic acids*, or *tannins*. They differ widely in chemical constitution and reaction, but all have the common property of precipitating gelatine and forming insoluble compounds with animal tissue. They are all compounds of carbon, hydrogen, and oxygen, but their ultimate structure is little understood.

When heated, the natural tannins yield catechol $C_6H_4(OH)_2$, or pyrogallol $C_3H_3(OH)_3$, and sometimes both; this has found a basis of classification. The classification of tannins on these chemical lines bears out an old classification made on the appearance of the tannage.

7. They were formerly divided into tannins that yield the whitish deposit on the surface of the leather, called *bloom*, and those that do not. Most tannins that give a bloom to the leather are *pyrogallol tannins*; a solution of their non-tannins when separated has an opalescent appearance. The tannins that give no bloom to leather are classed as *catechol tannins*.

When treated with sulphuric acid, the pyrogallol tannins yield gallic acid or ellagic acid, the latter combining with tannic acid to form the bloom. Under this treatment the catechol tannins yield the reddish-brown insoluble bodies

known to all tanners as *reds*. These differ from the original tannins in containing fewer molecules of water, and are therefore anhydrides of their especial tannic acids.

Hemlock bark yields a series of these reds; the higher members are soluble, precipitate gelatine, and are the principal coloring materials of the bark. The lower members are insoluble at ordinary temperatures and form the sediments found in the tannery vats.

WOOD AND BARK EXTRACTS

8. General Remarks.—The use of bark and wood extracts in the process of tanning is becoming more general every year. There are few tanneries where extract is the exclusive tanning agent and but few tanneries where it is not used to some extent. Primarily, an extract is valuable because of the annual decrease of tan bark. It brings the strength of distant virgin forests to the aid of our own failing supply of bark. As a convenient means of quickly strengthening a weak liquor, without the addition of the same amount of insolubles that would be added were the ordinary bark liquor used, it is decidedly useful. The extracts made from some barks and woods are valuable for the color that they impart to inferior tannages on which they may be used.

In sole-leather manufacture, extracts are generally used for strengthening the liquors and occasionally to improve the color of the stock. In some branches of the upper-leather industry, the pelts are partially tanned in bark liquors and then finished in liquors made up entirely from extracts, thereby imparting the distinctive characteristics of the tannin of the bark or wood from which the extract was made.

9. Manufacture of Extracts.—This business is carried on to a large extent in this country and of late years in South America. The material to be extracted is ground fine and the leaching takes place in the same manner

as at the tannery; details as to the duration of the leaching, temperatures, etc. being determined by the particular substance undergoing extraction. The liquor is then treated by some sort of clarification process, either mechanical or chemical; or both, to remove fine insoluble materials.

Numerous chemical methods of clarification are in use among extract manufacturers. Dried blood and other albuminous products are made use of, which in coagulating envelop the solid particles and settle out. Inorganic processes, such as the use of acetate of lead, or the precipitation of barium sulphate directly in the tank, and many other ways have been devised for purifying, but all remove more or less tannin in addition to the insolubles taken out. Mechanical processes of clarification consist in the settling of the liquor in vast settling tanks or by slow filtration through some coarse fabric.

After clarification, the liquor is pumped to a vacuum pan, where it is reduced in volume by evaporation until the desired consistency is reached. Some extracts are marketed with a specific gravity of 1.22, or 44° Twaddell, others are still further reduced to a specific gravity of 1.26, or 52° Twaddell, and lately solid extracts containing 10 to 15 per cent. water have appeared.

Hemlock-bark extract has been longest on the market. It is made from the common hemlock bark in localities where hemlock is common and shipped to other points not so favored.

Rock-oak bark, chestnut-oak bark and wood, sumac, quercitron, mimosa, quebracho, gambier, and other woods and barks are extracted, and their extracts form valuable adjuncts to the ordinary tan liquor.

Quebracho extract is gaining in popularity with tanners. It is made from the wood of the quebracho tree, a close-fibered wood that grows in South America. It produces a fine-colored tannage, but is better adapted to the tanning of upper leather than for sole, as its low non-tannin content tends to keep it "sweet," i. e., does not foster the growth of organic acids necessary to plumping.

LEACHING OF BARK

10. General Remarks.—The improper leaching of bark is an operation in tanning by which thousands of dollars have been allowed to go to waste. Even at this late day tanneries are leaving in the bark as high as 25 per cent. of its original tannin content. When the theory of leaching is understood, there should be no excuse for this wanton waste.

The principles of saturation and equalization underlie the theory of leaching; a piece of hot iron placed in water loses its heat to the water and becomes colder, while the water is warmed by the iron until finally both iron and water are of the same temperature. It is similar with leaching; both the leach and liquor become of equal strength. A strong liquor run on a spent leach will come off the leach weaker than when it ran on. In a perfect leaching system, the object is to always run a liquor on a leach that can add tannin to the liquor.

11. Preparation of Bark for Leaching. — Various methods of preparing the bark for leaching are in use. They all consist of forms of grinding and shredding, and each form has its particular disciples. This preparation is quite important and essential to close leaching; the best leaching system will not leach well if the bark is not reduced to such a state that the water or liquor can readily penetrate to every fiber.

Bark mills for grinding bark have the common feature of a revolving disk into which the cutting knives are set radially. Most of them have a secondary disk, which completes the work the first disk started; the bark then passes through small apertures to the conveyer.

12. Ott Bark Mill.—The Ott mill, shown in Fig. 1, is the most popular of the grinders. It consists of a hopper *a*, leading to the lower part of the mill, where the grinding is done. A shaft *b*, by means of which the mill is driven, runs

through the center of the entire mill and is keyed so that the mill may be driven from either the top or the bottom. The portion of the shaft running through the hopper has four

FIG. 1

iron arms c keyed to it, which project, and with the aid of similar projections c_1 from the inner sides of the hopper, break the bark into small pieces, which then fall into the runner, or carrier, shown in Fig. 2 (a). The runner, or

carrier, consists of a circular piece of steel *d* fitting the lower part of the mill; it is attached to and revolves with the shaft that passes through its center. Attached to the shaft above this disk are four angular wings *e* of iron that extend to the edges of the disk and are attached to it by bolts engaging the flanges of the wings. The ends of the wings are supplied with steel tips *e'* that form a cutting surface. The knives *f*, Fig. 1, are set vertically and bolted in a slotted cylinder the height of the runner, and surrounding it. The bark, having been broken by

FIG. 2

the upper arms, falls into the runner and is ground by being carried against the knives by the steel-tipped wings. Passing through the knife slots, it falls into the bottom of the grinder and is delivered by means of a spout *g* to the carrier *h*, which takes it to the leach house.

The cylinder, shown in Fig. 2 (*b*), containing the knives *f*, is in segments, each segment containing five knives, so that they can be removed for the grinding and setting of the knives. The mill is run at a speed of 100 to 150 revolutions per minute; the knives require grinding and setting about twice a week.

Many tanneries prefer a shaving machine; this reduces the bark to shreds instead of grinding it. With oak bark it certainly gives better results than the ordinary grinder.

13. Williams Patent Bark Shredder. — This form, shown in Fig. 3, is the best known of this type of machine. It is cylindrical in shape, the casing *a* surrounding a horizontal shaft to which a hub *b* is keyed. Heavy sharp-pointed arms, or hammers *c*, are hinged to the hub by pinions, and when the shaft is in motion the centrifugal force causes them to radiate from the hub. The arms, or hammers, are sharp pointed and present a cutting edge. The hopper *d* is at the side, the floor of the hopper being

FIG. 3

inclined by a raised plate, the end of which forms a sharp breaking corner so that the bark is cut in small pieces before the final shredding takes place. A segment of a cylinder, shown at the left in Fig. 3, contains the knives *e* and is placed beneath the revolving arms so that the edges of the knives and the sharp ends of the revolving arms form shears and cut or shred the bark forced between them. The shredded bark then falls between the knives into the conveyers. The segment, or "cage," containing the knives is in the form

of a semicircle, and the knives are so arranged that the lip, or projecting edge, presents a shredding surface instead of a pulverizing surface. The knives can be taken from the cage, resharpened, and replaced. A bowed steel shell *f*, with one edge riveted to the top of the casing and the other edge projecting through the hopper opening into the cylinder, forms a chamber for the settling of the dust after it has been moistened by a spray of steam introduced at the side of the hopper at *g*. Economy of power with greater grinding capacity is claimed for this type of machine; it is especially valuable for wood and barks of a fibrous nature.

When ground, the bark is conveyed to the leach house by some form of mechanical conveyer or blower, the former being the cheaper method of conveyance.

14. Vat Leaching.—Most of the older leach houses were constructed by sinking the vats in the ground; while this method seems to economize the heat used in latter-day leaching, the inability to repair leaks forms a serious objection, and the newer types of leach houses are all constructed with the bottoms of the vats above ground. In this way leakage is detected and stopped, and the spent tan is much more easily removed from the leaching vats. Mechanical contrivances for handling the bark before and after leaching are in general use. The leaches are filled from an overhead conveyer leading from the bark storehouse; and when the bark has been leached, the spent tan falls through an opening in the bottom of the vat into a conveyer that takes it to the furnace, where it is used as fuel.

The vats used for leaching are usually arranged and worked in set the vat containing the freshest bark is known as the *head leach* and that containing the nearly spent bark is called the *tail leach*.

15. The general scheme for leaching with vats is to run water on the tail leach, and after the liquor comes off it is put on the next, and so on throughout the set until the liquor comes off the head leach, when it is sent to the settling tanks or storehouse. The tail leach, after receiving all

the water intended for it, is emptied or "pitched," filled with fresh bark, and becomes the head leach, the leach next above it becoming in turn the tail leach.

This method of handling the liquors is common to all systems of vat leaching; it is in the method of applying the water, of removing the liquor from leach to leach, the temperature of the liquors, the depth of the vats, and in many other minor details that the various systems differ.

Some systems advocate the use of warm liquors on every leach, the water going on the tail leach being as near boiling as possible; this becomes cooler as it goes to the next leach and finally becomes too cool, in the judgment of the advocates of this system, and is reheated.

Some leaches have steam coils, protected from the bark by a false bottom, in order to maintain the temperature deemed necessary for proper leaching. Hot leaching, if not carried to extremes, is the most economical method; when the continued use of very hot waters is resorted to, the reds and the resinous matters of the bark are brought into the liquor. These are of no use in the tanning operations, and serve to make an impure liquor, their proportion to the amount of tannin that is removed by the extra heat being altogether too large to be economical.

The higher a column of bark through which a liquor passes, the stronger is the liquor and the more perfect the leaching; the length of the column is limited by its interference with the circulation of the liquor; when the leach is too deep, the bark packs down tightly and stops the even percolation of the liquor.

16. In some systems of leaching, the liquors are moved from leach to leach by pumps worked by a common rod. The conditions in each leach are, however, not the same, and separate pumping devices should be fitted to each leach so that the regulation of their speed is under control.

It is the practice in some tanneries to entirely empty a leach of liquor and then run on more liquor. The leaches should be kept covered with liquor at all times. If one is drained

free of liquor, the bark settles down and packs tightly; the liquor running on the bark when in this state channels its way to the bottom and even percolation is not secured.

A constant and slow influx of liquor on the leach is preferable to a large stream. The liquor should be given a long contact with the bark instead of being rushed through the leach. It should be delivered on the top of the leach in a divided stream and not in a single strong jet.

Partially spent liquors are frequently returned to the leach house in order to be strengthened and also to keep up the general strength of the leach-house liquors. These liquors have lost a considerable part of their tannin during the tanning operations but still retain a large proportion of the original non-tannin content; when strengthened, therefore, they do not represent the tannin strength that a new liquor of the same specific gravity possesses.

A complete description of a leaching system will give a general idea as to the mode of operating; the leaches are circular in form, 12 feet in diameter, and 15 feet deep, having false perforated bottoms, under which are steam coils. The temperature of the liquor going on the head leach is about 140° F., while that of the water on the tail leach is as near 212° F. as possible. The bark is carefully packed, care being observed that it is not packed too tightly. Two hot waters are run on the tail leach, the leaches being kept full.

17. Drum Leaching.—Leaching systems involving the use of drums have been the most successful. The apparatus of the Vaughn system is shown in Fig. 4. The drums *d*, situated at the floor level, are driven by the belt *a*. The dry tan bark is delivered to the drums by the conveyer *c* and the spouts *b*. The conveyer *e* is for removing the spent tan. The pumps *f* are for handling the liquor; *g* is the ground line. In this system, the bark is placed in drums and the water run in, the doors closed, and the drums revolved, thus bringing the water in constant contact with every particle of bark and also agitating the contents so that a liquor of uniform density is always present in the drum. The laws of

saturation and equalization must always be observed, and after the liquor has reached a certain point of saturation it must be changed and run on stronger bark, weaker liquor taking its place in the first drum. Closer leaching in shorter time can be effected in this manner than in any other.

18. Density, or Specific Gravity, of Tan Liquors. The density of tan liquors is determined by means of a hydrometer with a graduated scale showing the "degrees barkometer"; this form of hydrometer is called a **barkometer**. One degree barkometer represents the density of a liquor having the specific gravity of 1.001; barkometer degrees being the decimal portion of the specific gravity figure read in thousandths, thus: 10° Bark. is specific gravity 1.010; 45° Bark. is specific gravity 1.045, etc.

A barkometer merely shows the density of a liquor or its relative weight to water; it cannot determine the tannin strength of a liquor, as one 20°-Bark. tan liquor might contain 3 per cent. of tannin and another liquor of the same density might only contain 1.5 per cent. of tannin. Non-tannins and other soluble constituents of tan liquors influence the density, and hence the mistake should not be made of considering a 25°-Bark. liquor necessarily stronger in tannin than a 20°-Bark. liquor. The barkometer readings should be made with the liquor at a temperature of 60° F., or else a correction should be made to compensate for the expansion or contraction of the liquor. If warmer than 60°, the liquor will show less barkometer strength; if colder, it will show more, so that all readings should be made at, or corrected to, 60° F.

The specific gravity of an extract is generally denoted in degrees Twaddell. One degree Twaddell is equivalent to a specific gravity of 1.005, being one-fifth of the decimal portion of the specific-gravity figure when the latter is represented in thousandths. Thus, specific gravity 1.225 is 45° Twaddell; specific gravity 1.400 is 80° Twaddell, etc. From this relation, it is evident that 5° Bark. are equal to 1° Twaddell, or 45° Twaddell is 225° Bark.

THE STRUCTURE OF ANIMAL SKIN

19. In structure, an animal skin is made up of several readily defined layers. The skin is not merely a covering for the animal, but also serves as a container for nerves, secretive glands, and as a bed, or ground, for the hair, or fur. It is made up of two principal layers: the *epidermis*, or *cuticle*, and the *corium*, or *true skin*; these layers are totally distinct in structure, origin, and functions; under these two layers lies the under skin.

The *epidermis* is a thin covering of the true skin and is entirely removed before the tanning operations begin. The upper, or external, part is in life being constantly worn away, falling off in the form of dry scales. The interior part of the epidermis is made up of numerous living nucleated cells; as the surface of this layer is reached, these cells become flattened and finally dry up to form the exterior part of the epidermis and ultimately drop off as dead scales. These cells, which renew the horny external tissue of the epidermis, are in turn renewed by vessels situated in the corium, or true skin. The epidermis does not combine with tannin and is removed, laying bare the true skin, which is known to the tanner as the **grain**.

The *corium*, or *true skin*, is the skin with which the tanning agents combine to form leather. There is an intermediate layer of the corium next the epidermis; both layers are composed of interlaced bundles of fiber running across and above one another and parallel to the surface of the skin. They contain and are surrounded by fluid matter, which serves to keep the skin in a moist and pliable condition. Soaking in water removes this fluid and the fibers alone remain. These are saturated with the soaking water, and if dried by a gentle heat assume a horny appearance and become in substance about one-third of the raw hide.

The corium determines the quality of the leather that can be made from a hide. Its thickness, flexibility, and strength are reproduced in the leather. Between the fibers of the corium is an albuminous substance called *corlin*; in a dry state this holds and connects the fibers.

The connective tissue of the corium gives the form to the skin, the intercellular substance, coriin, lying between the fibers. When boiled with water, these connective tissue fibers form glue, losing their organized structure, and making a homogeneous jelly.

The intercellular substance is probably a decomposition product of the connective tissue. This coriin is soluble in lime water and in baryta water; frequent treatment of the skin with these reagents withdraws fresh quantities of soluble matter with every treatment. This fact should be noted in connection with the liming of skins to remove the hair.

Dilute inorganic acids swell the connective tissue, while strong mineral acids dissolve it. The swelling of the skin by acids is made use of to facilitate tanning; it not only makes the skin more sensitive for the reception of the tanning material, but also effects the disintegration of the fiber bundles into individual fibers, thereby increasing the surface.

The under skin is made up of loose connective tissue, imbedded in which are the sweat and fat glands, the blood vessels, and muscular fiber. It is removed in the beam-house work and plays no part in the tanning process; the side of the hide upon which this connective tissue of the lower skin is located is known to the tanner as the *flesh side*.

Fig. 5 shows an enlarged microscopic transverse

FIG. 5

section of the skin: *d* is the Malpighian net, or lower layer of the epidermis; *b* is the corium; *m* are lobules of adipose tissue; *a'*, the external orifices of the sweat glands; *y* is a hair follicle; *z*, a shaft of hair in the hair follicle; *i*, hair papilla. At *l* is the connective tissue of the under skin, showing the sweat glands *a*, with the ducts *c*, through which the sweat passes to the surface of the skin; *k* is a hair bulb and at *e* are openings of the sebaceous glands. The horny outside layer of the epidermis *h* shows at different places, as at *f*, hair or wool; the hair is not embedded immediately in the surface, but in the capsules, or sacs, being continuous with the epidermis and reaching down to the corium.

THE MANUFACTURE OF SOLE LEATHER

TREATMENT OF THE HIDES

20. Soaking.—Whatever may be the class of hide in use at the tannery, the first operation is that of **soaking**. This is always done by means of water, with or without the addition of chemicals. The various kinds of hides require different treatment as to mechanical handling, character of the soak, and the duration of the soaking. Whether the hide is “green” or has been dried with preservatives (in this case these preserving agents must be washed out), the dirt, blood, and dung must be removed, and the hide brought back to a state similar to that in which it was when taken from the carcass. All this foreign matter goes into the soaks, and it may be easily seen that but a short time elapses before the water becomes filthy.

Where hard water is used, it is customary to add some softening agent, as borax, since soft water is better adapted to soaking purposes. Much valuable hide substance is lost in many tanneries by allowing the use of soaks that have become putrid; an old soak gives better results than a new one, but if the vat is allowed to become putrid, depletion of

the hide takes place, and the loss from this cause more than offsets the time gained. Plenty of pure water with a little borax, if needed, is a much more effectual cleanser and softener than a putrid liquor.

21. The most general form of receptacle for the soaking is a pit or vat sunk in the ground. Wheels or drums have been used, and in some places are still used, but the ordinary vat is the most convenient for the purpose. The hides are either hung in the water on racks or thrown in and spread out. Dried hides are generally placed in piles and the water then run on them.

While in the soaks the hides must be watched by skilled eyes and hands so that they do not become putrid. Thin hides must be removed as soon as they become soft, without waiting for the thicker ones. The thin parts of all hides must be carefully watched, in order that they will not become damaged while waiting for the heavier parts to soften.

22. Hide Mills.—These machines are used where heavy hides are being made into sole leather. A hide mill is merely a heavy iron shoe on the end of a vertical beam keyed at the other end. A to-and-fro motion is imparted to the beam by a heavy shaft and the shoe moves in a heavy receptacle containing the hides. The hides are constantly in motion and in contact in running water, and a couple of hours of this treatment is more beneficial than a day's quiet soaking. The hides are first put in the soaks until they become softened, and the mills then used to finish the operation. Similar, but lighter, mechanical contrivances are used for the same purpose where lighter pelts are treated.

23. Depilation.—The unhairing, or depilation, of hides is most commonly accomplished by placing them in a solution of lime until the hair bulb is loosened, thereby allowing the hair to be readily scraped off. The lime acts on the roots of the hair, the hair sheaths are dissolved, but the hair itself is but slightly altered. The lime also acts on

the skin; the fibers swell and absorb water, so that the skins become plump and swollen, and at the same time the substance that glues the fibers together is dissolved and the skin becomes a mass of finer fibers. The lime also saponifies the natural fat, or oil, of the skin.

The use of lime is, however, unsatisfactory and more or less wasteful, owing to its energetic action on animal tissues; more or less of the gelatinous tissue that would form leather being lost, and unless the lime is removed from the hide after it has done its work, the leather will be hard and brittle and will be difficult to tan. The brittleness may also be caused by the saponification of the fats and oil originally in the skin.

Lime vats are made of timber or of masonry and are sunk into the ground so that the tops of the vats are on a level with the floor. The contents of the vats are made up by slaking the desired amount of quicklime and thoroughly plunging the vats. Generally two or more lime vats of different ages are used, the hides remaining in one vat for a time and then being moved by hand reels between the vats to the next lime vat.

The time required for liming depends on the kind and condition of skins, conditions of the atmosphere, and the temperature of the limes in use.

The question when to throw away the limes is also an open one; some tanners use them until they are almost saturated with decomposing matter extracted from previous hides. A lime in this state becomes a depilator by means of putrefaction and not because of its caustic action.

Lime water has been proposed as a substitute for the milk of lime in use; it is milder in its action, but also less permanent and requires frequent renewals in order to insure the perfect cleansing of the hides. Lime must be present in the vats so that a saturated solution of the hydrate of calcium is always surrounding the hides. Some claim that the presence of solid lime is essential; this is difficult to disprove because of the slight solubility of the hydrate, and because the lime in solution is rapidly

consumed in the depilation of the hide. Much of the residue of the lime vats, generally mistaken for unused lime, is in reality carbonate of lime formed during the liming process. However, it is found practical to have an excess of lime in the vats so that frequent plungings will keep the lime liquor saturated.

Sulphide of soda is sometimes used with the lime; it forms caustic soda and calcium sulphide when it comes in contact with the lime. The calcium sulphide acts as a solvent for the hair itself and renders easier the removal of the very fine hair. Because of its cost, sulphide of soda has not obtained a very wide use in sole-leather tanneries.

All the metallic sulphides are depilatories; the arsenic and sodium salts find frequent use in unhairing fine hides, from which limp leathers are made. In a general way the process of unhairing these light hides is carried out in a manner similar to the depilation of heavy leather, the time required is not so long, besides, it is shortened by the presence of the sulphides. In some cases the depilatory is spread over the hair side of the skins, and allowed to do its work in this manner. Other modifications of the manner of unhairing will be treated in the descriptions of the special tanning processes under which they occur.

24. Sweating.—In removing the hair by what is known as the sweat process, the hair is loosened by putrefaction of the hair sacs. In this country, the cold-sweat system is generally used, while the warm-sweat method is employed abroad. Dry flint hides from Eastern countries are generally prepared by sweating; they must first be thoroughly soaked and all parts softened. For this purpose the heavy hide mills are valuable adjuncts to the soak pits. Experienced men are employed to carefully watch the hides while sweating, as no hard-and-fast rules can govern their handling.

25. Cold-Sweat System.—This system as used in this country is carried out as follows: The sweat pits are vaults built of stone with the proper apertures for ventilation, in

order to regulate the temperature. Tight doors are provided, and these may also be used for ventilating purposes. After the hides are thoroughly soaked and softened, they are hung over poles or suspended by the edges in the sweat pit. The doors are closed until the putrefaction begins, and when this takes place careful watching is necessary. They are carefully scrutinized, and those that are further advanced in the putrefaction must be removed at once. The presence of a hide in the sweat pit for even a few hours after it is ready to come out may spoil it.

The sweating process hastens the expansion of the hide, opens the pores, and brings the hide back to a state analogous to that in which it was at the time of slaughter; it gives to the dry, wild hide a much-needed development. During the process much ammonia is generated as a result of the putrefaction taking place. Efforts are being made to isolate the specific germ which causes the putrefaction that loosens the hair; if they are successful and an antiseptic found that will be fatal to germs other than these, the sweating process will be greatly simplified.

After sweating, the hides are thrown into water or a weak lime bath, which stops further putrefaction.

26. Warm-Sweat System.—This system as practiced in Germany consists in placing the folded hides in an airtight buried box, which is then covered with spent tan. Steam is frequently used to accelerate the sweating process, but its use is attended with great risk, and the spontaneous heat generated by the hides is preferable. This system requires even more care than the cold-sweat process, as putrefaction promoted by heat makes rapid progress and may cause great loss.

If fresh-slaughtered hides are to be depilated by the sweating process, the flesh side is thoroughly salted and the hide then folded; they are piled one upon another and covered with straw. They are taken down after 18 or 20 hours, the hides opened and refolded, and again piled and covered, after which only occasional turning is needed until the hair

begins to slip. Most of the fresh-salted hides in this country are limed and not sweated.

After the liming or sweating operation is completed the hides are ready to have the loosened hair scraped off and to have the fleshy and fatty matter on the flesh side removed. These operations are known, respectively, as *unhairing* and *fleshing*.

The unhairing and fleshing of hides is done both by hand and by machine. The latter method is slowly superseding the hand method, being retarded because of the care needed in operating the machine. An unskilful or careless operator may spoil hundreds of dollars' worth of hides in a day's work, and the greatest care and judgment must be exercised on the part of the workman while running the machine.

The operations of unhairing and fleshing take place in what is known as the **beam house**. After the hair on the hides has been loosened they are brought into the beam house; if the sweat pit has been used to loosen the hair, the hides are placed in cold water, which cools them and interrupts the putrefaction that has set in. Limed stock is generally unhaired without previous washing, being brought directly from the limes to the beam house.

27. Unhairing.—The beams on which the hand process takes place are convex slabs of stone, one end resting on the ground while the other is about the height of the workman's waist. Over these sloping stones the hides are spread and by means of a concave knife, the curvature of which conforms to that of the beam, the hair is removed. The operation is fatiguing, and only men of long experience, skill, and considerable strength are employed.

The knife is pushed against the hair, the sides being worked from the hind shank towards the head and then towards the back. Occasionally very fine sand is used to facilitate the removal of the hair from places where it sticks. In removing the short, fine hair, hides are occasionally damaged by having the grain cut; to prevent this, a

guard is sometimes used, which projects beyond the edge of the knife and serves to protect the hide.

After the hair is removed, the hides are placed in water and are usually ready for fleshing the next day.

28. Fleshing.—The fleshing operation by hand consists in removing the fleshy and fatty matter by means of a sharp knife, and requires great skill. Both straight and curved knives are used in the operation. The hide is thrown across the beam, which has first been covered by a hide already fleshed. The flesh is removed by long strokes of the knife, which is held slanting, the right hand being uppermost. A long, steady drive results in fewer accidents to the hide than a short, choppy stroke. After the flesh has been removed from the main body of the hide, the hide is trimmed with a sharp knife and the shreds and projecting filaments are removed. All the fleshings are saved and sold for glue making.

After fleshing, the hides are hung on individual sticks in vats of fresh water. If the hides are to be used for upper leather, the soaking is dispensed with and the hides at once bated.

The hand method of fleshing and unhairing is too slow, too expensive, and involves too much disagreeable labor to last much longer. Machinery in the hands of skilled operators now does the work at a cost approximately one-third that of the hand process.

29. Machine Fleshing.—With machinery, the hides are fleshed either after soaking and before liming or after they come from the limes. If fleshed before liming, they are generally run through the machine again after coming from the limes, in order to remove the flesh left on after the first fleshing, and also the flesh raised up by the liming process.

The machines that do the beam-house work are heavy, rigid affairs. The removal of the hair and flesh are accomplished by rapidly revolving rolls, having raised irregular spines on their surface. The two most common types are the Vaughn and the Whitney machines.

The hides are fed to the Vaughn machine, shown in Fig. 6, by placing them on a feeding table *a*, which resembles a longitudinal section of a cylinder; by means of a lever this bed is set in motion and is carried towards the revolving fluted and spined roll *r*, bringing the hide in contact with it. A soft-rubber cushion *c* serves to protect the hide while it is being worked. When the hide has been worked through the cylinder, the bed is returned to its original position and the operator may reverse its position and again run it through.

The Whitney machine unhairs the hide when it is feeding into the machine and works it when it is feeding out. In operating this machine, the hide is laid on a traveling rubber apron that feeds it to two feed-rolls, which carry it to the working cylinder having slate blades or spines. After the hide has passed half way through the machine, the feed is reversed and the hide is fed out into the hands of the operator, who turns it and works the other half in the same manner as the first.

By the substitution of rolls with different forms of blades, a variety of work may be done by these beam-house machines. When the hides come from the machine, they are generally gone over by a workman, who trims edges and removes any hair or flesh that the machine has skipped.

30. Bating the Skins.—This process is also called *drenching* or *puring*. To effect the removal of lime left in the skins, the use of **bates**, or **drenches**, is resorted to. The most primitive bate, and one that is widely used today, is that made from the excrement of pigeons, chickens, or dogs. A fermented infusion of bran is also quite commonly used as a drench.

By the fermentive action of the bates, organic acids are formed which make soluble lime compounds that can be readily removed by washing. It has been found that the action of such bates is bacterial, and recent research work by German chemists has resulted in the isolation of the bacteria forming the ferment.

While in the putrefying liquor, putrefaction begins in the skins, and the fat, dirt, and undergrowth of hair become loosened and are easily removed. Bating causes more or less depletion of the hides; the putrefaction causes the waste of animal tissue and sometimes results in the loss of the entire skin. The bate is not under control, and the hides in it must be carefully watched in order that the putrefaction will not be carried too far to the detriment of the grain of the skin.

The **dung bate** is prepared by placing the finely divided dung in vats filled with water and allowing the infusion to stand for several weeks at a constant temperature. The upper part of the liquor is taken for use. The skins are either placed in revolving drums containing the bate or are put in revolving wheels having small openings and which dip in a vat containing the bate as they revolve.

Various attempts have been made to procure substitutes for these disgusting bates. The problem is to secure an organic acid that will form a soluble lime salt, which will not deplete the skin, further fermentation, nor cause plumping. Hydrochloric acid will readily remove the lime, but mineral acids cause the hides to become swollen. A mixture of old cheese, glucose, and ammonium chloride was formerly used; this causes a lactic-acid ferment. Commercial lactic acid is becoming prominent as a bate. Naphthalene-sulphonic acid is the main constituent of a patented bate that finds quite a market in this country.

Bates are used only on hides that are to be made into pliable leathers. They are seldom used in making sole leather, as these hides are placed in weak, sour, tan liquors, the lactic and acetic acids present accomplishing the same result as a bate, and at the same time plump the hide, preparing it for a stronger solution of tannin.

31. Plumping the Hides.—In sole leather, where weight is desired, it is advantageous to secure a good degree of plumpness before the leather reaches the liquors that really do the tanning. By **plumping** is meant the swelling of

the cells and the distension of the pores, so that the entrance of the tanning liquor is facilitated. If the hide becomes too plump, hard, brittle leather results; hence it is very essential that the proper degree of plumpness be attained.

The plumping is done by acids that are either formed in the tan liquors or are added to them. The natural acids formed in a vegetable tan liquor are lactic and acetic. Both of them are of bacterial origin, formed by the decomposition of the sugars forming a part of the non-tannins of the bark. The presence of small particles of hide substance in the liquor furnishes the nitrogen that nourishes the bacteria. If nitrogen is not present, very little acid is found. The absence of the requisite amounts of non-tannins in the liquors will also cause a scant supply of plumping acids. For this reason several so-called *sweet tanning agents* are unfitted for tanning heavy leathers, unless mixed with other liquors rich in non-tannins.

Quebracho liquor is a liquor in which the proportion of the tannins to the non-tannins is so great that acid is slowly formed, and a quick tannage is secured, the leather not being well filled nor nourished. If quebracho is mixed with some liquor with a large non-tannin content, this difficulty is removed. The extracts made from oak or chestnut woods are great acid makers because of the high percentage of non-tannins that they contain.

32. Methods of Obtaining Plumping Acids.—In a well-regulated yard the acid formed will be in the proper proportion to the tannin in the liquor, and there is no need of the addition of plumping agents. It sometimes happens, during the cold seasons of the year or when a yard is starting up after an extensive shutdown, that the acid is deficient. Various methods are resorted to in order to help the formation or supply of acid. Crude molasses is sometimes added; a mixture of old cheese, molasses, or glucose, and some nitrogen-containing substance, as ammonium nitrate, will stimulate the production of lactic acid. These are

generally kept in vats, called **rockers**, or **handlers**, so that they will not reach the rest of the yard.

On the other hand, it sometimes happens that too much acid is formed in the yard. The reason for this is not so easily determined. It may be because of the season or the weather; it may be caused by the method of leaching, the nature of the water supply, or from other causes that might seem too petty to produce such an effect. A tannery leaching its bark closely will have more acid in the yard than the tannery that is not so economical in the leach house. A very soft water or a peaty water will help acid production.

A yard with too much acid is a harder proposition to handle than a yard with too little acid. It requires close watching to prevent the production of hard, cracky leather and a consequent heavy financial loss. This condition of the yard may be only temporary or it may be a permanent one. In correcting too great acidity, the handlers, or rockers, that first receive the green stock should receive the first treatment. Dilution with weak sweet liquor, or even with water, will help their condition. Soda added to them will, of course, neutralize the acid, but care must be taken as to the amount added; too much soda will make the stock too soft.

The addition of an antiseptic, as corrosive sublimate or carbolic acid, will stop the formation of acid, but will not lessen its amount. The former is the better antiseptic to use, as carbolic acid sometimes tends to darken and spot the leather. Since the antiseptics do not decrease the amount of actual acid present, they are best used in the yard vats to retard the development of acid in that quarter.

In a properly managed sole-leather yard, where bark liquors are used, the deficiency or excess of acid is unusual. Some tanners pay no attention to this matter, depending on acids introduced into the first liquors for plumping. Commercial acetic and lactic acids are commonly used, and while they accomplish the end for which they are put in, they are unnecessary in a sole-leather yard, where natural means will generally furnish the acid required.

Where the commercial acids are used, continual vigilance must be exercised in paying attention to their strength and the amounts to be used, otherwise the hides will not be uniformly plumped, and the leather will show a corresponding non-uniformity of texture and fineness. With certain classes of hides used in making a special class of sole leather, sulphuric acid is used for plumping; the use of this agent will be discussed in the description of the manufacture of this leather.

33. The plumping is done in the handlers, or rockers—vats containing liquors that have been weakened by usage and are well soured. The acids present affect the plumping, and the small quantity of tannin in the liquors serves to partly tan the exterior portions of the hides. To equalize the action of the liquors in these vats, the hides are handled, or rocked, so that all portions of the hide come in contact with the liquor, and the liquor itself is constantly in motion.

The handlers, or rockers, are generally arranged in sets of a number of vats varying in strength. In handling the hides, it was formerly the custom to lift each hide from the vat and throw it into the next vat in the set, this process continuing through all the vats in the set. It is usual now to tie the hides one to the other, and by means of a hand reel the continuous string of hides is moved from one vat to the other. The hand reel is a wooden-stripped cylinder mounted on a stand about 4 feet high and turned by a crank. In the operation, the reel is placed between two vats, one man turning the crank while another starts the string of hides over the reel and then arranges them in the vat that receives them, the friction of the hides on the reel carrying them along.

Handling by rockers is done by means of a wooden frame hung by pivots in the center of the top of the vats, so as to give a dipping movement of 6 to 8 inches to each end of the frame. The hides are hung over sticks placed across the frame. Motion is imparted to the frames of the rockers by beams connected with overhead shafting. By this means all

parts of the hide are kept in contact with the liquor and the liquor is well agitated. This end is also obtained in some tanneries by hanging the hides in the vats and agitating the liquor by means of an air pump, which supplies air to the bottom of the vat. The air bubbling up through the liquor keeps it stirred.

TANNING SOLE LEATHER

34. General Remarks.—The simplest and most typical methods of tanning are those used in the manufacture of sole leather; the methods of tanning other leathers are mainly variations of these. For this reason, a discussion of the various processes of tanning sole leather will precede those for the tanning of the more delicate ones.

The system at present pursued in the tannage of sole leather is the result of an evolutionary process depending on the selective ability of the tanners themselves. No scientific discoveries have helped them, and the basic principles of their art have never received much attention. This state of things is due to several causes: In the first place, in an art that has obtained a high degree of perfection, through the endeavors of generations of practical workers, we are apt to rest with the results achieved without questioning the principles involved. In the second place, the raw materials, hide and tannin, being organic bodies of an unknown, or at least a very complex, nature, there is no foundation for theories of their natural interactions until more knowledge of them is obtained. The methods that have resulted from the evolutionary process of practice serve admirably for the purpose, and by means of them the cost of production is kept so low that the field is not an inviting one for the prospecting scientist.

35. Gain.—Sole leather is always sold by weight, and the cost of the hide is from 50 to 70 per cent. of that of the finished product. The object of the tanner is to make as many pounds of leather from a given weight of hides as he

can possibly produce. His degree of success in this is technically known as **gain**. In the case of green-salted hides a large percentage of the hide, as it is bought, consists of water and salt, and such impurities as dirt, dung, and blood, so that 100 pounds of green salted hides will only make from 60 to 80 pounds of finished leather. The gain would be spoken of as a gain of from 60 to 80. Dry hides contain practically nothing but dried hide substance, and 100 pounds of this class of hides will make from 150 to 185 pounds of leather, the gain in this case being a gain of from 50 to 85.

With green hides, the **gain** is the amount of leather made from 100 pounds of hides; with dry hides, the gain is the amount of leather made from 100 pounds of hide, less 100.

Clean, dry hide is worth at the present figures about 35 cents per pound; tannin pure and dry in the liquor figures about 6 cents per pound. It is therefore essential to get as little of the former and as much of the latter into the finished product as the leather will bear.

36. The process of vegetable tannage is no doubt a chemical one to a certain extent, but after the final chemical reaction between the hide and the tannin has taken place, the product is capable of a further absorption of tannin; the point at which chemical reaction ends and physical absorption begins has not been determined. Suffice it to say that sole leather may contain from 20 to 50 per cent. of tannin, and in all cases it will be thoroughly cured and perfectly tanned. There is, however, a minimum limit at which hide treated with tannin is leather, and this limit is the satisfaction of the purely chemical affinity of the hide for tannin.

The whole process is an economical one; the tannin does not change materially while in solution, and it is almost completely recovered from the tan liquors by the hide during the process of tanning. Labor costs are low; the only disadvantage being the time consumed and the concomitant loss of interest.

There are three general varieties of sole leather made in this country, the names given them indicating the bark used in tanning. They are *oak*, *hemlock*, and a combination of both, known as *union*; each of these classes of leather has special modifications producing special grades of leather. The greater part of the sole leather made in this country is tanned by hemlock bark; the European prejudice against this leather has been removed and immense quantities are annually exported. Hemlock leather is produced by two methods of tanning—*acid* and *non-acid*; some *slaughter hemlock* is also made, but this forms only a small percentage of the aggregate output.

OAK-BARK TANNING

37. Oak Leather.—Leather tanned with oak bark is distinguished by its color and texture; oak bark gives a deposit, or *bloom*, of ellagic-tannic acid on the leather, and *oak leather* is known as *scoured*, or *Texas*, depending on the removal of this bloom.

The hides used are green and generally of native slaughter. The Texas hides are usually from range cattle, are branded, and are made mostly into unscoured leather; those that are not branded or have only small brands are used for scoured leather. South American hides make a good grained scoured leather and are in demand for the finest class of men's shoes.

The hides are placed in the soaks for about 3 days, but the time may be shortened by using warm water and a loss in hide substance thereby saved. The water in the soaks is changed with each pack of hides; some tanners remove the hides after the first day's soaking and replace the dirty water with fresh.

After soaking, the hides may be fleshed or they may be put in the limes and the fleshing and unhairing done after they leave the limes. Different tanners have different methods for liming this class of hides; some leave them in the limes for as long as 5 days, others complete the liming in 3 days, handling the hides continually with frequent limes. It is

claimed that a warm bath of, say, 100° to 110° F. given to the hides the night previous to unhairing is beneficial.

After unhairing and fleshing, it is the custom in some tanneries to bate the stock with molasses bate; with the amount of acid usually present in an oak tannery, this is unnecessary and is the exception rather than the rule. The hides are usually placed in a vat of clear water and remain there over night. The hides are then suspended in the rockers and the real tanning begins. The object of the rocker liquors is the removal of the lime remaining in the grain (it being neutralized by the organic acids in the liquor), the swelling of the hide, and the setting of the color. The quality of the leather is made in the rockers and handlers according as the plumping, coloring, etc. is properly done.

The liquor used in the rockers should be old, mellow, and not strong; the strength of the liquor is regulated by the number of pits in use, so that the green hides should have sufficient acid to purge it of lime and properly start the plumping.

From the rockers the hides go to the handlers, remaining there from 8 to 14 days, according to the system in vogue at the tannery. Some tanners allow the hides to remain in the same liquor for the required period, while by others the liquor is regularly replaced by stronger liquor. It is quite usual also to handle the hides from one vat to the other, starting with the tail handler, or weakest liquor, and continuing until the head handler, or strongest liquor, is reached.

The handler liquors are stronger than rocker liquors, the strength being governed by the extent to which the tanning has progressed in the rockers.

38. Lay-Aways, or Layers.—From the handlers the hides are taken to the main tanning yard, where the lay-away vats are located. These vats are generally 9 feet long, 7 feet wide, and 8 feet deep. In these the hides are laid out flat one by one and a thin layer of ground oak bark sprinkled

over each hide; this bark should not be ground too fine, or the weight of the pack will cause the hides to settle together too much and the circulation of the liquor will be prevented, thereby destroying one of the purposes of the bark, and ultimately resulting in imperfectly tanned leather. Each layer is treated in the same way, fresh tan or bark being used in each case.

After laying away has been begun the tanning is conducted on lines peculiar to every tanner; each man has his own ideas as to how much liquor the hide should receive and under what conditions. In general the hides are allowed to remain in a lay-away until the liquor in that vat is sapped; the hides are then removed, the liquor run off, and stronger liquor used after repacking the hides as before.

Each pack of hides is laid away from 4 to 7 times, according to the theories and judgment of different tanners, the whole operation of laying away extending over a period of from 4 to 5 or 5½ months. These successive lay-aways are called **layers** and are known as first, second, third, etc. layers, according as they are the first, second, third laying away that the hides have received.

39. Tanners differ not only as to the number of layers that oak leather should receive, but also in regard to the temperature and strength of the liquor on these layers. For example, the following schedule is in effect in a large oak tannery:

TABLE I

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer
First.....	18	30
Second.....	22	32
Third.....	30	35
Fourth.....	35	40
Fifth.....	55	45

For light hides only four layers are given in this tannery, the liquors being slightly weaker except the last layer, which is almost as strong as that used on heavy hides; the time required for tanning the light hides is, of course, shorter. In this tannery no attention is paid to the temperature of the liquors beyond keeping them at a working temperature of 65° to 70° F.

The hides go from the handlers to the first layer; this liquor is an old liquor, although not very sour, strengthened either by extract or by returning it to the leach house and strengthening it there by running it over a leach of bark. After the first-layer liquor is sapped, the pack is raised and placed into the second layer; the second-layer liquor is a sweet-bark liquor, its strength not requiring the addition of extract. The third-layer liquor is also a sweet-bark liquor that may or may not be strengthened with extract. Fourth-layer liquor is also a sweet liquor, with the addition of extract. The fifth and last layer in this tannery is also a sweet liquor, bark extract being used freely to bring it up to the required strength.

It is in the last layer that the leather receives most of the physical absorption of tannin and non-tannins; a strong liquor applied for a protracted period always benefits the leather. If the hides have received the proper treatment up to the last layer and the last layer is strong, with good circulation, a well-nourished leather results, one that is literally full of bark.

40. In another successful oak tannery, six and sometimes seven layers are given; the temperatures of the various layers are also kept well up, and if it is necessary to tan the leather in a shorter period, these increased temperatures, with proper regard for the strength of the liquors, will produce the desired effect. It has been only a few years since the inauguration of warm liquors in the yard, and they have not been well received by all tanners. The schedule in this yard is as follows:

TABLE II

Layer	Time in Layer. Days	Strength of Layer. Degrees Barkometer	Temperature. Degrees Fahrenheit
First....	14	38	70
Second..	20	34	75
Third. .	30	34	80
Fourth..	30	38	90
Fifth....	45	42	100
Sixth....	50	50	110

The time taken for tanning by this system is a little longer than by the first method, but the leather seems to be of a closer texture, better filled, and of better wear-resisting qualities. The liquors are made up in practically the same way, although there is not so much extract used. The first layer is an old liquor, the others being new, sweet liquors, and with the exception of the fifth and sixth layers no extract is used. For these layers a large proportion of oak-bark extract is used, being mixed with the heated liquors and the mixture being then cooled to the proper temperature of the layer.

41. Oiling and Drying the Leather.—After tanning, the hides are taken from the last layer and split into sides; after rinsing, the sides are piled to drain over night and then swabbed with cod oil, a mixture of cod and fish oils, or a mixture of mineral and fish oils; the swab is generally made of sheepskin. The sides are then hung on sticks or by brass catches in the drying loft.

The drying loft is generally located over the vat yard; light is excluded, but a proper degree of heat and ventilation must be maintained. It is usual to effect this by mechanical means, blowers carrying air to the loft and keeping the atmosphere in constant circulation. If there is not proper circulation, the air becomes stagnant among the closely hung leather and the damp leather will become moldy;

apertures, which may be regulated as to size, are generally distributed around the floor of the loft and aid in keeping the air in circulation. This end may also be attained by forcing air into the tightly closed loft, so that the pressure is slightly above atmospheric, this means effectually moving any stagnant air.

The leather is hung over poles whose ends rest on beams, or it may be held by an ingenious brass device in which the weight of the side causes the friction to uphold it.

42. Rolling the Leather.—When thoroughly dry, the sides are taken from the drying loft to the rolling room and there brushed with water and made into covered piles; when properly tempered the sides are rolled and dried in the loft, and when dry are sorted for the market. The rolling of the leather serves to smooth the grain side and also to put somewhat of a polish on the finished leather; it is effected by machinery.

43. Leather Roller.—This machine consists of a heavy beam having on its end a polished brass wheel about 8 inches in diameter and with a 6-inch face; a crank gives to the beam a direct motion, allowing the brass wheel to move about 1 foot each way right and left. The leather is held under the moving wheel, or roller, on a small bed of iron conforming to the arc described by the roller; a foot lever serves to raise this bed and makes the pressure of the moving roller light or heavy, according to the will of the operator; by moving the leather around under the roller all parts of it are finally smoothed and polished.

In some tanneries, the vat sizes of which necessitate it, the hides are split into sides as they come out of the soaks or out of the limes. Whole-hide tannage is more economical as to labor costs in the yard, but side tannage is more economical in beam-house work.

A large, light-colored, and even deposit of bloom on oak leather is considered a mark of fine tannage; for some purposes, the leather is sold with the bloom removed, known as

scoured leather. For this leather the hides are split and rinsed as usual and sometimes milled for a time; then they are scoured by the scouring machine, drained, oiled, and dried; they are then dipped in water, rolled, and dried, and then receive a final rolling before going to the sorting room.

NON-ACID HEMLOCK TANNING

44. In the **non-acid hemlock process** of tanning, the hides are swelled, or plumped, by the natural ferment acids in the tan liquors; while in the acid process, a solution of sulphuric acid is used to effect the plumping.

45. Treatment of the Hides.—The hides used are generally dried South American hides; they are well soaked and the hair loosened by the sweating process. After the removal of the hair, the hides are soaked over night and are then placed in the handlers. The strength of the handlers and the subsequent tanning liquors depends on the tanner and his methods of leaching; when the leaching is close, the liquors are generally of high barkometer strength, although the proportion of tannin to the total solids in the liquor is not so great as in the case where the leaching is not so close. The handlers are arranged in sets of vats that contain liquors of different strengths, the tail, or weakest, handler being about 10° to 16° barkometer. The hides are started in the tail handlers and successively pass through the series, the handlers serving the purpose of coloring and plumping the hides.

When the hides are sufficiently colored or plumped they go to the main tan yard to complete the tanning. Here they receive from five to seven layers in liquors of different strengths, according to the methods in use at the tannery.

46. A schedule in use at a successful non-acid tannery is as follows:

TABLE III

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer
First	18	28
Second	18	30
Third	24	32
Fourth	40	34
Fifth	45	36

In this tannery the liquors are used at a temperature of from 70° to 75° F. on all the layers. The first layer is an old liquor that has been sapped by usage on the last, or next to the last, layer. In the second layer is a sweet liquor, the last layer sapped is used for the first layer; but if this last layer is to be used without strengthening, it is used for the second layer, and the next to the last layer when sapped becomes the first layer.

The hides are kept in the first-layer liquor for the required number of days; the pack is then hoisted, the old liquor removed for handler purposes, and the hides placed into the second layer; this process of treating the skins to successively stronger tanning liquors goes on until the last layer is reached; the leather is then finished as far as its actual tanning is concerned. The length of time for each layer is a matter determined by the judgment of the tanner, and therefore differs with different conditions. Theoretically, it would be best to allow a pack of hides to remain in a layer until there was no more sappage, or taking up, of tannin by the hides. There is no question but that some of the periods of time on the first two or three layers are too long; after the tannin content of hide and liquor is equal, months of further contact will not put any more tannin in the hide in that layer.

The third, fourth, and fifth layers are new, sweet, bark liquors, coming direct from the leach house. In some tanneries hemlock-bark extract is used to strengthen these layers.

47. Use of Warm Liquors.—There is some prejudice against the use of warm liquors in a tan yard, but if the proper limit of heat is not passed, the results seem to be beneficial. By their use, the length of time on the beginning layers is shortened and stronger liquors used. In one tannery, where warm liquors are used, seven layers of liquor are given to the stock. The schedule of this tannery is as follows:

TABLE IV

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer	Temperature. Degrees Fahrenheit
First....	16	30	70
Second..	18	32	80
Third....	18	34	85
Fourth..	20	34	90
Fifth....	20	36	100
Sixth....	30	36	110
Seventh.	40	38	120

The liquors at this tannery are, with the exception of the first-layer liquor, sweet bark liquors. The time on the last two layers is occasionally cut down if the hides are light, and in the case of very light hides the last layer is dispensed with. The temperatures on the different layers is adhered to within a range of 5° either way. With these warm liquors the duration of the tanning process may be shortened, but an increased period does not harm the stock.

48. After leaving the last layer of tanning liquor, the hides are split into sides; this is done in some tanneries prior to this. The sides are then drained, oiled, and dried in the drying room. After drying they are wet down and then rolled, after which they are ready for the market.

Some hemlock leather is treated to a bleach of quebracho and borax after leaving the last layer and then sold as union-tanned leather. Various methods of bleaching and weighting the leather with foreign matter have been resorted to, but such sophistications are not common at present.

ACID HEMLOCK TANNING

49. Acid Hemlock Leather.—This leather is a variety of hemlock sole leather that has been plumped by means of sulphuric acid. Other forms of sole leather are plumped by organic acids, either naturally present in the liquors or added to them; the sulphuric acid causes an abnormal distension of the pores and swelling of the tissues.

Most of the acid hemlock leather is made from dried hides; that made from green hides is known as *slaughter hemlock sole*, the process being the same, except that the hides, being green, are depilated by means of lime instead of sweating. The dried hides are largely of Asiatic origin, this being a very popular method of tanning the Chinese "Buffalo" hides.

The hides are soaked, sweated, depilated, and fleshed in the usual manner and are soaked in clear water. They then go into a weak tanning liquor called the *coloring vat*. This exerts its astringent effect on the hides to a slight degree, and they are slightly colored by the bath.

50. Plumping With Sulphuric Acid.—After the above treatment, the hides are put into a .1- to .3-per-cent. solution of sulphuric acid and remain in it until the swelling has progressed to the desired stage; they are then ready for the tan yard.

Because of the abnormal plumping caused by the sulphuric acid, the hides can safely be put into strong liquors. In the other processes of sole-leather tanning the hides receive weak-liquor treatment, the strength being gradually raised until the maximum strength is attained; in this branch of the industry there are no true rockers, or handlers. The only tannin treatment they receive before going to the first-layer liquor is the slight coloring gotten from the weak tanning solution before plumping.

After plumping, the hides go to the first-layer liquors and are given the time required for this layer. The first-layer liquor in an acid tannery soon becomes impregnated with sulphuric acid and sulphates and must then be discarded. The free sulphuric-acid content is generally not very high

in this liquor, but there is sufficient to interfere with the tanning action. The liquors become very dense and the proportion of the tannin to the total solids is low, so that there is little sapping to be obtained; the gravity of this first layer sapped, or "run-to-creek," liquor is from 18° to 25° Bark. and it will contain from .8 to 1.5 per cent. of tannin.

To save the tannin and also to be rid of the necessity of running this strong filth-bearing liquor into a stream, has occasioned some experimenting. It has been proposed to remove the sulphates by precipitation with barium chloride and subsequently settling. In some tanneries it is the practice to run this liquor over a spent leach of tan bark; this removes the free acid and some of the solid impurities.

51. As in the case of the other sole-leather processes, there are various systems in use in this branch of the industry. There is a little more latitude here, however, than in oak or non-acid hemlock tanning, the first-layer liquor being used in many strengths by different tanners. Very little extract is used by acid tanners; they prefer to run the hides through bark liquors as quickly as is consistent with a good gain. From five to seven layers of liquors are used, either cold or warm. A schedule of strengths in a cold-liquor tannery is given in Table V.

TABLE V

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer
First	14	38
Second.....	20	40
Third.....	20	42
Fourth.....	25	42
Fifth	35	44
Sixth.....	50	48

The first-layer liquor is a sixth-layer liquor that has been sapped by a tan pack. The other liquors are all new, sweet

bark liquors run from the leach house. The temperature is kept about 65° F.

52. In a warm-liquor tannery using seven layers of liquors, the schedule is as follows:

TABLE VI

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer	Temperature. Degrees Fahrenheit
First....	12	41	65
Second..	22	42	75
Third...	24	43	85
Fourth..	24	44	90
Fifth....	24	46	100
Sixth....	30	48	105
Seventh.	45	49	110

In this tannery, the first-layer liquor is a sixth-layer liquor that has been sapped; the second layer is a seventh-layer liquor that has been sapped; the other liquors are sweet bark liquors made without extract.

After coming from the last layer the hides are drained, and if they are to be bleached are placed in vats of cold water. After bleaching the leather is allowed to drain and is then swabbed with the oil in use at the tannery. They are then placed in the drying loft, and when dry are wet down and rolled and again taken to the loft.

53. Acid slaughter sole is made from green hides, the hair being removed by liming. A bate is sometimes used on this variety of leather, but otherwise the liquors and operations are the same as in the manufacture of the regular acid hemlock. The use of sulphuric acid as a plumping agent results in the production of a firmer leather. The action of the acid is much more energetic on sweated hides than on limed stock, so that in the regular acid process care must be observed as to the degree of plumpness attained.

UNION TANNING

54. Union Leather.—The combination of hemlock and oak barks gives a very serviceable leather known as **union sole**. It is of a better texture than hemlock, is harder, and has a color approaching that of oak. There is probably more variation in the different processes of making this leather than in the other kinds of sole leather.

The color is all important, and many methods are in use for its improvement; some go back to the leach house in aid of color, and sacrifice tannin there in order to have very pure liquors, which will give the leather a bright, clean shade. An illustration of the wide difference in union methods may be seen in the fact that in some union tanneries the first-layer liquors are as low as 8° Bark., while in others they run as high as 25° Bark. These liquors are not of the same purity, however; the 8° liquor will show a higher proportion of tannin to the total solids than the 25° liquor, but aside from this the tannin strength of the 25° liquor is greater than that of 8° liquor. In tanneries where strong liquors are used, considerable oak or chestnut-oak extract is used, the upper layers being strengthened with it, and the texture and color thereby improved.

55. Most of the union leather made is **cropped**; that is, the bellies and shanks are cut from the sides, making the product in the form of rectangular pieces of leather. The crop leather thus made commands a higher price than union side leather, because it can be used without so much waste when being cut into soles. The bellies, shanks, etc. are sold for scrap leather for inner soling, etc. A good quality of cut soles is made from union leather, and the method of selling these cut soles determines the method of tannage in some tanneries; a certain number of soles will be cut from several tannages of leather, and the leather that will give the largest number of soles to a given weight of leather is generally the best seller, if other considerations, as color, firmness, and texture, are equal. This causes tanners to endeavor to produce a light crop leather of a good color,

strength, and closeness of texture. This can be obtained by the use of very pure liquors of low barkometer degree. The purity of the liquor being secured at the expense of leaching, it is questionable whether this method pays, after all. More leather can be made from a ton of bark by this process, but the actual gains will not be so great as if a heavy, well-filled leather is produced.

Green, salted hides are used for making union leather; they are well soaked, limed, unhaired, and fleshed, and are then placed in clear water over night. They then go into the rockers and handlers; according to the method of tanning in use, these and all the other liquors vary in strength. After coming from the handlers, the leather is cropped in some tanneries, while in others it is tanned and finished as side leather and then cropped. From the handlers, the sides go into the lay-away yard; from five to seven layers are given, using oak bark on every layer. The oak bark is ground fine, but not fine enough to prevent circulation of the liquor between the sides in the vats. After use in the lay-aways, the bark is sent to the leach house and leached with the hemlock bark for the union liquors; it is generally placed on the bottom of a leach, the hemlock bark being run in on top of it.

56. A schedule of layers and liquors in a tannery using cold, weak liquors is as follows:

TABLE VII

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer
First.....	18	14
Second.....	18	16
Third.....	20	18
Fourth.....	24	20
Fifth.. ..	30	23
Sixth.....	45	25

The leather from this tannery is of a very beautiful color, but light in weight; it is tanned quickly; the sapped liquors are strengthened by returning to the leach house, and no extract is used. The liquors themselves are very pure, some of them of such purity that 65 per cent. of the total solids are tannins; with this purity, the spent tan, of course, shows considerable tannin, it sometimes running as high as 2.25 per cent. The sides are completely tanned here and are cropped after finishing. The yard operations are carried on in the usual manner, the sapped upper layers returning to do duty as lower layers, being strengthened in the leach house.

57. To show the difference in methods, the schedule of another cold-liquor tannery is appended:

TABLE VIII

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer
First.....	14	24
Second.....	18	26
Third.....	24	26
Fourth.....	30	30
Fifth.....	50	34

The leather is of a good color, improved by slightly bleaching, and is well nourished. The spent tan shows little tannin and the liquors are not so pure; extract is used to some extent on the upper layers.

58. A warm-liquor tannery using strong liquors and considerable extract had the schedule in Table IX.

Six layers were in use at the tannery, although ordinarily but five are given. This tannery makes rather heavy leather, of a good color, and their bark is well leached. Extract is used for strengthening the liquors, and some are also returned to the leach house for strengthening.

TABLE IX

Layer	Time in Layer. Days	Strength of Liquor. Degrees Barkometer	Temperature. Degrees Fahrenheit
First.....	14	22	65
Second....	18	24	75
Third.....	22	26	80
Fourth....	26	28	90
Fifth.....	30	31	100
Sixth.....	40	34	105

The aggregate sapping of the liquors in all these strong-liquor tanneries is, of course, greater than in the weak-liquor process. The sapping is found by taking the degree barkometer of the layer before the pack is put in and again when the pack is raised, the difference between these two figures being the sapping on that layer. The sum of the sappings on the different layers gives the aggregate sapping. This shows the amount of tannins, which influence the barkometer, that have entered the leather. In some of the weak-liquor tanneries this aggregate sapping is as low as 18°, while in the stronger-liquor tanneries it runs as high as 35° or 40° Bark. The comparative purity of the liquors must, however, be considered before these figures are regarded as relatively absolute.

From the last layer the leather is raised and drained. If it is to be bleached, it is then put into vats of water until ready for bleaching. Some tanners improve the color of the leather after tanning by a bath of quebracho or quercitron liquor. The leather is drained from the last layer and then put into these extra liquors; a short time gives the desired effect. The leather is then drained, swabbed with oil, and dried. After drying it is brought to the rolling room, wet and tempered, and then well rolled. Sometimes an additional swabbing of oil or a solution of caseine is given and after a slight drying it is again rolled.

The leather after drying is sorted and cropped, if the cropping has not been previously done. When the cropping has been done out of the handlers, the bellies and heads must be tanned separately. They are generally tanned as quickly and as cheaply as possible. So much expense is entailed in handling these small and irregular pieces of hide that the process must be cheapened, in order to turn out the leather to sell at the market price and still leave a profit. Rather weaker liquors are used and more handling is done, the heads being tied to one another in long strings and moved from vat to vat by means of the hand wheel. The finished product is generally sold as rough leather without finishing or rolling.

THE MANUFACTURE OF UPPER LEATHER

59. General Remarks.—Upper and harness leather require a different treatment than sole leather, in order to produce a soft and pliable material. Upper, or light, leathers as they appear on the market may be whole hides, or they may be made by splitting heavy hides in a number of lighter pieces (called *splits*) for tanning.

If splits are to be made, the heavy hides are soaked, depilated, and fleshed in the usual manner, and generally bated, or pured. The hides are then partially tanned, generally in a bark tannage, until they are struck through with the tanning liquor.

When the tanning has progressed sufficiently for splitting, the hides are removed, allowed to drain, the surplus tanning material removed, and the hides *stoned* on a stoning jack to remove wrinkles so that the splitting machine may do its work properly. After being dried on poles in the tannery yard, they are brought in and wet down preparatory to splitting, which is done by machinery.

60. Splitting Machines.—Two general classes of **splitting machines** are in use, the Union and the belt knife.

The latter is the more popular machine and is found in most of the Eastern upper-leather tanneries.

In the Union, the leather is presented to the edge of the splitting knife by gripping and drawing rollers. The leather

FIG. 7

is held on the rollers by carrying arms and is split by the power by which the roller carrying it is rotated.

The belt-knife splitter, shown in Fig. 7 in front view, and in Fig. 8 in rear view, splits by means of an endless

belt knife *a*, against which the leather is carried by means of rolls. These machines are not automatic, and require skilled labor to operate them economically.

FIG. 6

When the hides are to be split, they are wet thoroughly and packed down until they are entirely moistened through. Each piece must be uniformly wet, for if a spot is drier than the rest of the hide, that spot will split differently.

A *skiving* is generally first taken off and the remainder of the hide smoothed out on a stoning jack before further splitting.

61. Stoning Jack.—The stoning jack, shown in Fig. 9, is built of iron and run by a side belt drive. By means of a crank-shaft, an arm *a*, supported and guided by a swinging truss, is given a to-and-fro motion, following the contour of



FIG. 9

an inclined metal bed *b*, the face of which is smooth and cut in the form of a segment of a circle. To the end of the arm is attached the "stone" *c*, a rectangular piece of metal with rounded corners and slightly longer than the width of the bed. When in operation this tool is carried backwards and forwards over the bed, at a distance from it regulated by

the operator by means of a foot lever. The leather is thrown on the face of the bed, under the moving arm, and the "stone" brought down on the leather; by its action it removes wrinkles and creases, the leather being meanwhile moved around under the stone by the operator, so that every part comes in contact with the smoothing tool. The grain side is finally split, making the split light or heavy, according to the future use of the leather.

62. The split is generally hand trimmed before further tanning. Some run them in a small wheel with weak liquor in order to remove the glaze caused by the splitting machine. After this they are put back in weak tanning liquor, which is gradually increased in strength until the split is completely tanned.

The grain, which has been almost tanned before it was split, is generally gone over by hand to remove any humps, and then the tanning is finished in a revolving drum with the tanning liquor. Sumac and quebracho are favorite tannages for this work, although the use of hemlock still obtains.

The finishing of the different classes of leather, resulting from the splitting, differs some, according to the part of the original hide from which the split portion has been taken. The grain side is generally buffed and the splits whitened.

63. Scouring.—There is little to be done in the finishing of sole leather, besides drying, oiling, and rolling; with upper, morocco, and other limp leather, the actual tanning is about half of the process of converting the hide into finished leather.

The rolling of sole leather was treated in the description of the process of making oak sole leather. For upper leather, **scouring** is employed to prepare the face of the leather, or set it out, so that it is fine, dry, even, and smooth. All the dirt, adhering tan, etc. are removed and the leather is thoroughly stretched, worked fine, and made firm.

The hand method of scouring is not so common as formerly. Tables sloping from the workmen are used; on these the

FIG. 10

leather is spread out, and by means of a short-bristled brush, a fine-grained flat stone, and a "slicker," the laborious operation is gone through.

Because of the labor involved and the expense entailed, the hand method has been largely superseded by machinery. The machine shown in Fig. 10 will scour, or set out, both upper and belting leathers. The skin is placed on the semi-circular table *b* and carried up under the revolving roller *a*, which is covered with blades so arranged as to work out the leather and stretch in both directions at once.

The pressure desired is at the control of the operator; the dirt, etc. worked out falls away from the machine, so that it cannot be again taken up by the leather.

64. Stuffing.—Stuffing is the treatment of leather in such a manner that it will contain the requisite amount of grease or oil as its future use may demand. It is effected by both hand and machine, largely by the latter method; the former is used only on certain classes of leather.

For stuffing leather, the oil par excellence is cod-liver oil. It has been in use since the early days of the industry, and as yet no other oil has been found that can satisfactorily displace it. Cod-liver oil has a good body and will not oxidize and form gums, so that it remains in practically the same state in which it entered and gives to the leather a permanent mellow feeling.

This oil is expensive, and therefore adulterations are common; seal, menhaden, and mineral oils are most commonly used as adulterants. Mineral oil may be detected by saponifying the fatty oils by boiling with potash, whereby the mineral oil, which will not saponify, will separate. Mineral oils may be distilled off at about 450° F.

It is almost impossible to detect for a certainty the fish oil when mixed with cod-liver oil. Fish oils have more gum than the genuine cod-liver oil; but cod-liver oil and menhaden oil have the same color and gravity and their range of odor and taste is about the same.

Neatsfoot oil is used with satisfaction if a good cold-tested oil is secured, otherwise a cold temperature will turn the skin white.

Sod oil is largely used for stuffing. It is expressed from skins, and after boiling and undergoing clarifying treatment is ready for use.

Degras is the oil obtained from wool during its cleansing process. French degreas is the surplus oil obtained in the manufacture of chamois leather. It is frequently adulterated, and comes on the market in all grades.

Glycerine is used by some tanners instead of degreas. It imparts to the leather a high degree of softness, but it should not be used alone, as it possesses a corrosive action and, being soluble in water, is easily removed from the leather by dampness.

Porpoise oil is much valued for stuffing.

After scouring, the sides are hardened in the air and are then dampened and tempered to facilitate the absorption of the grease; this process consumes from 1 to 3 days' time, depending on the manner of stuffing. If hand stuffed, the leather must be damper than if stuffed by the wheel.

65. Hand Stuffing.—This method of stuffing is passing away because of its expense and for other reasons. The leather must be so much damper for this method that a longer period is required for drying; after the absorption of the oils, the surface must be gone over to remove the unabsorbed matter; then, too, the stuffing, when done by hand, penetrates only slightly beyond the surface, and the leather is left dry and hard and practically unchanged.

This operation as practiced consists of rolling the sides into bundles with the grain side in and softening by heating; then the warm oil and tallow is applied to the flesh side with a brush.

66. Drum Stuffing.—Stuffing by this method is effected by means of a revolving drum *a*, shown in Fig. 11, into which the skins are placed to receive the stuffing. The skins are dampened thoroughly on both sides and allowed to

lie in a pile covered by damp cloths. . The drum is heated by steam or hot air for some time before receiving the skins. The heat is turned off and the skins placed in the drum and then run for some time with the door loosely closed. This loosens up the goods and regulates the temperature; the stuff is then run in hot, but not hot enough to scald. The tight door is put on the drum and the skins wheeled for from $\frac{1}{2}$ to 1 hour. The drum is stopped and the door opened and the drum slowly revolved for a few minutes. The skins are then taken out, smoothed, and laid in piles.

FIG. 11

They are generally stoned as soon as possible on marble or plate-glass tables (it is best to stone on the grain side first) and then hung up until nearly dry; the operation is repeated on the flesh side, removing all marks of the stone, and the sides are then hung up until quite dry. Several patented forms of stuffing drums are on the market, in which the heating is effected by means of coils containing steam or hot air. This keeps up the temperature of the drum while the stuffing is going on and does away with the water of condensation, occasioned by the direct presence of steam. A hot-air stuffing mill is shown in Fig. 12; the air for the drum *a* is heated by steam coils in the heater *b*.

The tannage of the goods and their class, or grade, determine the kind of stuffing to be used. For the best



FIG. 19

leather, cod-liver oil is generally used; for other and cheaper grades, various combinations of the oils and greases mentioned above are substituted.

THE FINAL FINISHING OF UPPER LEATHER

67. Whitening, or Buffing.—After stuffing and setting out, the next operation in the manufacture of upper leather is that of *whitening*, or *buffing*. This is done by hand, either on a table or on an inclined beam similar to the beam used in the beam house; or it may be done by machinery.

68. Whitening Machine.—Whitening by machinery is effected by a machine, shown in Fig. 13, consisting of a rapidly revolving cylinder on the end of an arm having a pendulum motion. The face of the cylinder is covered with parallel knives, or spines, running transversely. The leather

FIG. 13

is laid on a bedplate under the moving arm and the arm, with the cylinder, lowered by means of a foot treadle until it comes in contact with the leather. The pressure of the tool upon the leather is regulated by the operator, who keeps the leather moving so that one section of the surface

does not receive more of the abrasive action of the cylinder than the other.

The whitening machine illustrated is made of iron; the rotary cylinder, or whitening tool *a*, is driven by belts at either side running on a small countershaft, fixed in boxes at the end of the supporting double truss or pendulum *d*. The belt running the countershaft passes between the parts of the truss over a pulley at its upper end and thence to the main driving belt. The pitman *e* is connected with the balance wheel *f*, and from it receives the to-and-fro motion. The foot treadle *b* is for regulating the distance between the cylinder and bedplate *c*, and accordingly the pressure on the leather. The bedplate is of polished brass, slightly wider than the cylinder, and is inclined. A revolving emery wheel *g* travels constantly from one edge of the revolving cylinder to the other, keeping sharp the faces of the knives, or spines, of the cylinder.

69. Blackening. — After whitening, or buffing, the leather is ready for **blackening**. For the heavy grades of upper leather this operation is preferably done by hand. The liquid blacking or other dressing to be applied to the surface of the leather is spread evenly over the hide by means of oval brushes made of fine bristles; the excess of blacking is removed by other brushes, care being exercised that no blacking reaches the under side of the leather.

70. Blackening Machine.—The most popular machine for blackening leather is the Batchelder, shown in Fig. 14, or modifications of it. In this machine the leather is spread on the table *a* and fed into the machine, the edge of the leather being engaged between the surfaces of two rolls and then passed over a third roll, which forms a supporting bed for the skin, while the brush *b* applies the blacking. The blacking or other dressing is in the receptacle *c*, and the fluted roll *c'* running at the edge of the blacking receptacle supplies the brush with the coloring matter. The brush *d* seems to work the blacking into the leather and also to

remove any excess. After passing under the second brush over the supporting roll d' , it is delivered upon a moving bed e , formed by endless tapes extended about rollers f and f' and driven by gears.

As the hide does not always cover the roll supporting it under the blacking brush, some blacking reaches the roll; this roll must be kept clean, otherwise the under surface of a wide portion of hide would become soiled with the blacking; a wiper or cleaner of some flexible material, held by an adjustable bar against the lower surface of the roll, effects the cleaning. The material wiped, or scraped, from the roll drops into the trough g and passes to a bucket provided for that purpose.

The rows of bristles on the brush are so arranged that alternate rows incline in different directions, those of one row inclining towards one end of the cylinder and those of the next alternate row towards the opposite end. In this way the ends of the bristles, besides traveling over the leather in the direction of rotation of the brush roller, also, when in contact with the leather, spring in opposite directions longitudinally and thus work the blacking backwards and forwards, as well as in the direction of the movement of the roller, effectually blackening the leather and preventing streaking.

71. Surfacing.—The leather after being properly blacked is submitted to a **surfacing** process. This operation is sometimes performed by hand, but by far the greater part of the leather finished in this country is *glazed* or *pebbled* by machinery. The surfacing machine shown in Fig. 15 is similar in operation to the rolling machine used for sole leather, but is lighter in construction. The roller a runs in boxes on the end of a beam b connected directly to a wheel c on the drive shaft, which imparts a direct to-and-fro motion to the beam. The end of the beam is supported by a pivoted truss d , which also serves as a guide, so that the roller runs in a line parallel to the face of the bedplate e . The bed on which the leather is laid is inclined and its face

is straight. On all machines for finishing the surface of leather, every precaution is taken to guard against oil or grease coming in contact with the leather. A single grease

FIG. 15

spot will greatly depreciate the market value of a skin. The rolls are generally self-oiling and the boxes are especially devised to guard against oil leakage.

72. Glazing.—For glazing, a smooth roller is used, the leather being laid on the bed and moved around under the moving roll, so that every part of the surface receives

its polishing action. The heat essential to a good polish, or glaze, is supplied by friction of the roll, and the operator must be careful not to maintain too great a pressure on the leather, or the heat generated will burn the leather. The bed is controlled by screws in front of the machine, one screw raising or lowering both ends of the bed at the same time; one screw regulates the pressure for both ends. The other screws adjust the bed to any desired position.

An electrically heated roll has lately appeared on the market and has become quite popular. The roller is heated to the temperature necessary for a good polish, and pressure need not be resorted to in order to attain this end.

73. Pebbling.—For pebbling, dicing, graining, or for giving the leather any desired surface, different rolls are substituted for the smooth roll used for rolling and glazing. These rolls have their surfaces cut in the desired figure, and by the action of the machine the design is imparted to the surface of the leather.

74. Embossing. — For embossing leather, a heavier pressure, distributed over a larger area of leather, is needed. The embossing machine shown in Fig. 16 consists of two geared rolls *a* and *b*; the lower one *b* is smooth and forms a supporting surface for the leather that is to be embossed. The upper one *a* is smaller, and the design to be embossed is cut in its surface. The leather is fed between the moving rolls and receives the design from the upper roll; the pressure is regulated by hand wheels *c* and *d* at either end of the machine, and a foot treadle *e*, connected with a clutch *f* on the drive shaft, assures perfect control of the machine by the operator.

BLEACHING LEATHER

75. General Remarks.—Methods of bleaching leather vary with the leather to be bleached. In general, the bleaching of light leathers is accomplished by true oxidizing or bleaching agents, while the so-called bleaching of heavy

leather is really a treatment of the surface of the leather with tannin solvents.

It is always the endeavor of the tanner to produce as light a leather as possible, but circumstances may cause a dark color; the correction of this dark color simply deals with the surface of the leather, so that a bleached, or toned, leather may have the outward appearance of a light-colored, high-grade leather, while the interior is not modified by the bleaching or tanning process.

76. Use of Alkalies.—For bleaching heavy leather, different alkalies are used to dissolve some of the surface tannin from the grain of the leather, and the dissolving action of the alkali is checked by a weak acid solution whereby the alkali itself is neutralized.

A solution of borax is prepared and the sides are either milled in it or simply hung in vats containing the solution. After a short period the sides are removed and dipped in weak hydrochloric or sulphuric acid and finally in pure water.

Weak ammonia water can be used for bleaching, hydrochloric acid being the best acid to use in connection with it. The proportions of alkali and acid must be carefully worked out and the bleach tried on a small scale before using it on a large lot of leather. If the alkali is too strong or the sides are left in too long, too much of the surface tannin is removed, and it becomes flabby and soft. If the acid bath is too strong and the excess of acid is not thoroughly removed in the vat, the leather will darken along its lower sides while drying.

In connection with the above alkaline and acid treatment, solutions of sumac, quercitron, or quebracho are sometimes used. Sometimes they are milled with warm solutions of these tanning extracts, with or without the addition of tin chloride.

77. Use of Sodium Peroxide.—An article in a foreign journal describes a method of bleaching by means of sodium peroxide. Magnesium sulphate is dissolved in water and the sodium peroxide added to the solution. The leather to be

bleached is first washed with a very weak solution of acetic acid and the bleach then applied with a brush. This process is a true bleaching one, and differs in this way from the alkaline baths above described.

The most carefully tawed kid leather sometimes requires bleaching in order to produce delicate shades in the dyeing. Chamois leather is also frequently bleached to remove its characteristic yellow color, so that it will not interfere with its later dyeing.

78. Use of Sulphur Dioxide.—The bleaching is generally accomplished by sulphur dioxide, either in the gaseous form or in a solution in the form of sulphurous acid. Formerly the skins were put in the wet state into a closed chamber, in which sulphur was burned. The gaseous sulphur dioxide formed sulphurous acid with the water on the skins, and the bleaching was thus effected; the skins when taken out were at once placed in clean water. Skins bleached by this process had not a uniform color, even parts of the same skin being bleached more than the rest of the skin. Because of this trouble, whatever bleaching is now done by means of sulphur dioxide is carried out in weak solutions of the acid.

The acid is formed by leading the fumes of burning sulphur into lead-lined vats of water. The skins are suspended on sticks in properly diluted solutions of the acid until the proper color is attained; the skins are then washed in cold water. Sulphurous acid bleaching will not give a permanently light color, but in contact with the air the color again becomes dark.

79. Miscellaneous Bleaches.—Hydrogen peroxide is occasionally used for bleaching light leather; chlorine, in the form of hypochlorites, is also an effectual bleaching agent, but must be carefully used. When bleaching chamois and leathers of a similar nature, other oxidizing agents are used in connection with sulphurous acid. Potassium permanganate or some other vigorous oxidizer is used; a solution of potassium permanganate is rubbed into the leather, which becomes brown from the deposition of manganic

oxide by the decomposed permanganate. After being thoroughly worked, the leather is put in a solution of sulphurous acid, the oxide is dissolved, and the brown color entirely disappears, leaving the leather perfectly white. Kid leather not pure white can be made white and the yellow tint neutralized by a weak bath of water-soluble aniline.

DYEING LEATHER

80. General Remarks.—The aniline colors are well adapted for leather dyeing, and almost all of them are greedily absorbed by the leather. Water-soluble anilines are best, giving brighter and clearer shades; dyes soluble only in alcohol seem to injure the fine grain of the leather by the action of the alcohol.

The very large range of shades and tones makes the leather-dyer's task a hard one, and careful management is necessary to secure more than one lot of leather of exactly the same tint. The temperature and strength of the dye bath, the duration of the immersion, the nature of the mordant, and the nature of the tannage of the leather are all factors determining the resulting shade of the finished leather.

81. Methods of Application of Dyes.—The dyes are applied either by immersing the leather in the fluid or by applying the dye with a brush. Leather tanned dark is generally bleached before dyeing. The skins are all sorted according to their color; the lightest are used to be turned into delicate shades of leather. Inferior ones in point of color are colored green, black, or other heavy, dark colors.

When a mordant is used, the nature of the dye to be applied determines the mordant. In leather dyeing, iron sulphate, alum, sulphate of alumina, the tin and copper salts, tartar emetic, and numerous other salts are in use; weak Castile, olive-oil, or fig-oil soaps are largely used for leather mordanting.

The dye bath is generally a little over 100° F. in temperature; the goods are turned over by hand, care being taken

that no skin remains long at the bottom of the pile. In the *paddle method* of dyeing large paddles are used. This method saves labor, but wastes dye and does not give a uniform color; for this reason its use is generally confined to the common classes of leather.

Dyes made from the various natural dyewoods are faster to light and are not so easily rubbed off as the aniline dyes. They are more expensive than the aniline dyes and involve considerable trouble and apparatus in their extraction.

82. Dyeing of Tan Shoe Leather.—The leather used for the popular tan shoe of the present day is dyed with a combination dye. If calfskins are used, the bloom must be removed before dyeing.

The skins are first wheeled with fustic extract and extract of peach wood; then the dissolved aniline is added to the wheel, the temperature of the whole being kept at about 100° F. After they come from the wheel they are dipped in cold water and then wheeled for a short time with a weak solution of bichromate of potash. Sumac extract is sometimes used as a mordant in place of the fustic and peach wood.

TAWING, OR MINERAL TANNING

83. General Remarks.—For making some forms of white leather, leather for belt lacings, etc. and glove leather, certain agents are used that are quite different from any previously mentioned. These processes (employing mineral tanning agents) were originally known as *tawing*, but the term has since been enlarged, and now includes *chrome tanning*.

In **tawing**, yolks of eggs, flour, alum, salt, and sulphuric acid are used. The hide thus treated becomes tough, pliable, can be stuffed, and will not decompose. Mineral tannages using alum, salt, hyposulphite of soda, and iron salts have been introduced, but with the exception of alum and salt they have not attained any practical value.

84. Tawing Kid-Glove Leather.—As an illustration of tawing leather, the process for turning lambskins and

kid skins into glove leather might be described. The skins are thoroughly soaked in cold water for a couple of days and when soft are put into a revolving drum having wooden pins fixed about the interior to thoroughly shift the skins. Water is run through the drum while it revolves and thoroughly saturates the skins and removes the dirt. After washing they are limed in the lime pits, and the lime afterwards removed by running the skins over a paddle wheel. They are then depilated and fleshed by hand. After further treatment in the revolving drum, the skins are bated, or pured, in a mixture of bran and water. After being well washed after puring, they are put into the tanning drum with the liquor that is to do the tanning. This is generally composed of flour and the yolks of eggs, or salt and alum, or it may be a composition of three or all of these substances in varying proportions.

After running the drum from 12 to 15 hours, the skins are taken out and hung up to dry. When dry, they are dampened with water and softened, by squeezing and pressing by means of swinging planks and finally by drawing the skins individually over a broad steel knife; this also serves to remove any of the dry tawing composition that may adhere to the skin.

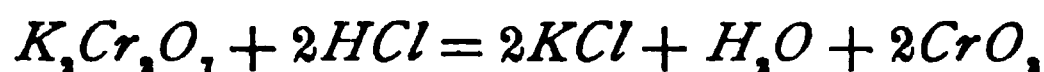
When ripe they are colored, being first washed in cold water and are then placed in a revolving drum in a bath of egg yolk. They are treated with a mordant consisting of a mixture of bichromate of potash and soda, and then colored by the addition of sulphates of iron, zinc, or alum, according as black, drab, or tan is desired.

85. Chrome Tanning.—A typical example of mineral tannage is the **chrome process**. This tannage is purely a chemical one, consisting of the combination of chromic oxide with the skin. Only from 3 to 6 per cent. of chromic oxide is deposited on the hide. This deposition is evidently a chemical combination between the skin and the oxide, as the product is not affected by washing or boiling, as are bark tannages; neither is there any deposition of the oxide

after the chemical union has taken place, even in the basic liquors used in one of the processes. All chrome leather made is tanned either by the *Schultz two-bath process* or the *Martin-Dennis one-bath method*.

86. Schultz Process.—This process consists in treating the skins to a bath of bichromate of potash in the presence of a mineral acid; following this, a bath of a solution of hyposulphite of soda is given. The theory of this process is based upon the formation of chromic oxide having the formula Cr_2O_3 , which is deposited on and combines with the skins. A solution of bichromate of potash and hydrochloric acid is made up in the proportion of about 5 pounds of bichromate and 2.5 pounds of hydrochloric acid, specific gravity 1.16, to every 100 pounds of wet prepared skins.

The reaction expected between the acid and the bichromate is



but in the process only two-thirds the acid necessary for the complete reaction is present, and it is not safe to say, considering the peculiar properties of chromium, that this formula represents the reaction.

After the hides have been in this bath they are put into a solution of hyposulphite of soda with a free acid; this effects the reduction of the CrO_3 to Cr_2O_3 . To neutralize the acid remaining in the skins, they are afterwards wheeled with water containing suspended calcium carbonate.

The use of practically all the agents that will reduce CrO_3 , as hydrogen sulphide, the sulphides and acid bisulphites, etc., has been patented; sulphurous acid seems to be the best, or at least it is the one most generally used. Norris has patented the use of zinc in the hypo bath, which, it is claimed, accomplishes a more rapid and economical reduction.

Amend has a two-bath process in which chromic acid H_2CrO_4 replaces the bichromate and an acid of the Schultz patent; this requires careful manipulation. He suggests the use of aniline as a reducing agent.

A chalk bath after the tanning bath neutralizes the hydrochloric and sulphurous acids remaining in the skins; solutions of sal soda and borax are also used for the same purpose. Norris has patented the use of potassium permanganate for this purpose; this salt not only oxidizes the sulphurous acid to sulphuric acid and neutralizes it at the same time, but it also precipitates the oxide of manganese on the skin, and this acts as a mordant in subsequent dyeing.

87. Glace Kid.—The manufacture of **glace kid** is a typical chrome tannage, and the larger part of it is made by the Schultz two-bath process; a description of its manufacture will also serve to illustrate the practical workings of this system of chrome tannage. Glace kid was formerly made exclusively in France, and was tanned with alum, salt, flour, and egg yolk, but the chrome-tanned article made in this country has displaced the foreign product.

The skins are well soaked and further softened by mechanical means; borax is generally added to the soaking water. After sufficient softening, they are placed in the limes, with which arsenic sulphide (red arsenic) has been mixed; this agent loosens the fine hairs better than any other. The skins are then unhaired by hand or machinery, thoroughly washed in borax water, and bated over night in a slightly heated bate made from dog or pigeon manure. Upon removal from the bate, the pelts are slated by hand or machinery, which effects the removal of the fine hair remaining on the skins. They are now put into a slightly heated drench of bran and water, and are finally washed with borax water.

The skins are now ready for the chrome bath, which is made up in the proportion of about 5 pounds of potassium bichromate and 2.5 pounds of hydrochloric acid, specific gravity 1.16, to every 100 pounds of wet hides. They are wheeled in a revolving drum with this solution until the yellow liquor has thoroughly penetrated the skins, and they have assumed an even yellow color throughout; this is determined by cutting pieces from the heaviest parts of the

skins and noting the appearance of the cut. When this liquor has penetrated the skins, they are removed and allowed to drain for a time and then placed in the reducing bath. This is composed of about 10 pounds of hyposulphite of soda with from 3 to 5 pounds of hydrochloric acid, specific gravity 1.16, for every 100 pounds of hides. Sulphuric acid may be used here in place of hydrochloric with equally good results. After wheeling in this solution, until a cut of the hide discloses a bluish-white color and a disappearance of the yellow color, the skins are regarded as tanned, and are then placed in the neutralizing bath of soda or chalk.

There is no hard-and-fast rule to follow as to the strength of the tanning baths; this depends on the class of the hide and is a matter of experience and judgment with the tanner.

The skins are finished in a variety of ways and colors and with a dull or glazed surface.

88. Dennis System.—This process of chrome tanning apparently offers less difficulty than the two-bath system, but it has not come into such general use; it seems to be better adapted to delicate skins than the Schultz process.

In the Dennis process but one bath is used; this is composed of a solution of a basic chrome salt that readily yields its excess of chromic oxide to the hide tissues. The tanning liquor used in this process is made by dissolving oxide of chromium in hydrochloric acid, thus forming the normal chloride; to this is added an alkaline base, as sal soda, and this stronger base takes from the chromium chloride part of its acid, leaving the oxy or basic salt. This basic salt is a very unstable compound and readily yields its excess of chromium to anything having any affinity for it, which in this case is the hide substance. The advantages of this system over the two-bath system are as follows :

1. One handling of the skins is saved because one bath only is needed for the tanning.
2. The skins are not exposed to the destructive action of chromic acid, and are therefore stronger, with more lasting suppleness, and have a better grain.

3. There are no offensive fumes evolved in this process; the reduction of the oxide in the two-bath process is accompanied by a strong odor of sulphur dioxide.

4. The skins are not harmed by too long contact with the tanning liquor.

5. Less skill is required to operate it and the bath itself is cheaper,

89. The practical operations of tanning by means of this system are as follows:

The calfskins or goatskins are soaked, limed, depilated, and fleshed as usual and placed in a bran drench. From the drench they are taken to a drum and milled with a strong solution of alum and salt. This keeps the skins open and plump and also has the effect of keeping them in good condition during and after the tanning. From the alum-and-salt bath they are put in the tanning bath; this contains a 3-per-cent. solution of the concentrated basic chrome liquor and also a liberal quantity of common salt. This common salt prevents the grain of the leather from drawing, under the astringent action of the chrome bath.

When the skins have assumed a uniform greenish-blue color and a cutting reveals the completion of the process, they are rinsed off in clean water and worked on the flesh side with a slicker on a perfectly clean table. The bath of suspended chalk may or may not be used, at the option of the tanner.

90. There are special and peculiar qualities of chrome leather that distinguish it from other kinds of leather, making it a superior article. Chrome leather is not actually waterproof, but rather a moisture resister; it is a difficult matter to thoroughly wet it when once perfectly dry.

The ordinary bark tannages are compounds of two organic materials, and when subjected to alternate wetting and drying will eventually deteriorate and become rotten; chrome leather, being a combination of an inorganic material with the hide substance, is not affected by this process

of wetting and drying. It is lighter than bark leather and also possesses more elasticity and is of greater strength.

91. The **finishing** or **currying** of chrome leather is carried out with some modifications of the methods for finishing bark leather. Chrome leather will not carry as much grease as the bark tannage; it must be stuffed soon after removal from the tanning bath and must not be allowed to become dry. The most efficient way of stuffing chrome leathers is by "fat liquoring"; there are different ways of making and using these fat liquors, according to the quality of the leather. The most common fat liquor is an emulsion of neatsfoot oil with a potash soot; it is used warm and applied in a revolving drum. Emulsions of egg yolk and degreas, or egg yolk and olive oil, are frequently used. Chrome leather is lubricated and nourished better by these fat liquors than by the hard greases and heavy fish oils that are used in bark-leather stuffing.

THE MANUFACTURE OF PATENT AND ENAMELED LEATHER

92. Preliminary Treatment.—In the manufacture of **patent** and **enamel** leather, selected heavy Swiss hides are used. The soaking, unhairing, fleshing, and liming are practically the same as for sole leather. They are then bated, and after being washed are placed in the handlers or rockers in weak tan liquors. The handlers are generally hemlock or oak liquors. After the hides are well struck with tannin, which generally takes from 10 to 14 days, they are taken out, the liquor pressed out, and the hides partially dried. They are then ready for the splitting machine; generally there are three splits made from the hide. After leaving the splitting machines, the splits are retanned. Hemlock may be used for this purpose, but generally gambier, japonica, or quebracho is used. These last are quick tannages, and as weight is not sought after in this class of

leather, a quick, light tanning agent is used. Quebracho in the extract form is becoming very generally used for this purpose and gives excellent results.

After this retanning the hides and splits are hand scoured on a table, to remove all excess of tanning material, dirt, etc. and to effect a "setting out." After being stuffed they are tacked on stretching frames and allowed to dry in a room heated by steam. When dry they are softened first by machinery and finally by hand. The goods are then sorted according to quality and size, different qualities and sizes being used for different purposes.

Enameled leather is grained or pebbled in order to give it a broken surface. Patent leather has a smooth, glossy surface. All grades of enameled and patent leather are not treated in the same manner in applying the composition. In general treatment, however, the lines are close and a description of the process for one grade will give some idea as to the method.

93. Enameled Leather.—The hides, splits, and buffings that have been selected for enameling are stretched and tacked on frames. They then receive a heavy coat of linseed oil that has been boiled down almost to a jelly. It is quite important that this daub should be properly made; generally 2 days are consumed in making it. The oil is boiled in a large kettle over a coke fire, being constantly stirred. The first day the temperature of the oil is run up to nearly 600° F. and then lowered to about 380° F. before it is left for the day. The second day the temperature is raised a little over 600° F. and the cooking continued until the proper consistency is attained. The oil is gradually cooled, the fire extinguished, and naphtha added in the proportion of 1½ gallons of naphtha to every gallon of oil. The naphtha is added in small portions with constant stirring. If the daub is to be the foundation for a dark color, the highest grade of lampblack is added under constant stirring.

After the leather has received a heavy coat of this daub, carefully applied and the excess removed, it is exposed to

the air until it is thoroughly dry. When dry a second coat is applied, and this second coat dried by placing the leather in ovens heated to about 120° F. The leather then receives another coat of boiled oil, the oil used for this coat not being so thick as that used for the two previous ones; after this coat has been dried in the oven, it is given a smooth surface by rubbing with pumice stone.

A coat of varnish made from boiled linseed oil and the blue or black pigment desired is then applied and the leather again placed in the oven. This varnish is reduced by naphtha in the same manner as the daub used for the first two coats. When the first varnish coat is dry, the leather is pumiced, the final coat of varnish applied, and the leather again placed in the oven; this time the oven is a little hotter than for the previous dryings.

After the sticky surface is removed by exposure to the sun and air, the finished leather is taken from the frames and hung in a damp room to regain its normal moisture. After trimming and graining by hand, it is ready for the market.

94. Patent Leather.—For patent leather, the split receives three coats of daub, the last of which is pumiced after it has dried; then the first coat of varnish is applied, dried, and pumiced. The second coat receives the same treatment. The daub applied is about the same as that used in enameling hides. The varnish used is made by boiling linseed oil with raw umber for several hours and then adding lampblack and reducing with naphtha.

Three coats of finishing varnish are then applied to the split, and after each coat has been dried in an oven at about 175° F. the leather is pumiced; with each succeeding coat a finer stone is used and the pumicing becomes lighter. After the third coat the leather is cleansed from all dirt, dust, and grit, and the final coat of Chinese blue varnish carefully applied; after oven drying, it is exposed to the air, removed from the frame, and is ready for the market.

THE MEASUREMENT OF LEATHER

95. General Remarks.—Sole leather is always sold by weight, but light leathers are sold by area, the approximate thickness also being taken into consideration. The measurement of leather is now usually done by means of machines, of which there are several on the market. Some of these vary essentially in principle, but the descriptions of the Sawyer and Union measuring machines given here will be sufficient to show their application.

FIG. 17

96. Sawyer Measuring Machine.—This machine, shown in Fig. 17, is run by power; the leather is fed between two rollers that smooth out the leather and bring

it into contact with small measuring wheels; the movement of these wheels influences properly arranged levers, which, in turn, affect the index of the registering mechanism.

97. Union Measuring Machine.—This machine, shown in Fig. 18, consists of a table with a top made of parallel

FIG. 18

sticks of wood; a hinged cover closes over this and throughout the entire surface of this cover are suspended small wires that are free to slide up through holes in the cover. They are arranged in rows, so that when the cover is down, with nothing between, the pins hang between the strips of wood forming the lower table.

When leather is to be measured, it is placed on these strips and the top brought down over it. The surface of

the leather obstructs the small pins and throws them up, the ends of the remaining wires dropping between the strips. These pins are of such sizes and weights that each exercises the same force on a spring balance above, so that this device is affected by the number of pins obstructed by the leather. The weight of forty-eight of these wires corresponds to a square foot of area, and the dial of the balance indicates, in fractions of a square foot, the weight released by the leather, and consequently the area of the leather. A machine that measures the leather in all three dimensions has been recently patented, but as yet has not reached the market.

ANALYSIS OF TANNING MATERIALS

98. General Remarks.—The methods of analysis for tanning materials have been gradually developed and are peculiar to this class of work. In addition to the general laboratory equipment of apparatus and chemicals, there are special contrivances and preparations needed, such as extraction apparatus, devices for shaking, animal charcoal, and a supply of hide powder.

ANALYSIS OF BARK, WOOD, ETC.

99. Preparation and Extraction of Sample.—The sample is finely ground, well mixed, and a 10-gram sample dried over night in a steam oven for moisture determination. A weighed portion of the bark or wood is placed in some form of extractor. The best extractor, shown in Fig. 19, is a modification of the well-known Soxhlet extractor; it is made of spun brass and is in two main parts. The lower can *a* is about 7 inches in diameter and serves as a boiler. The upper can *b* is 4 inches in diameter and has a taper bottom fitted with a perforated brass plug *g* that makes a close joint with the neck of the lower can. The top of the upper can is flanged and fitted with iron clamps *f* that fit

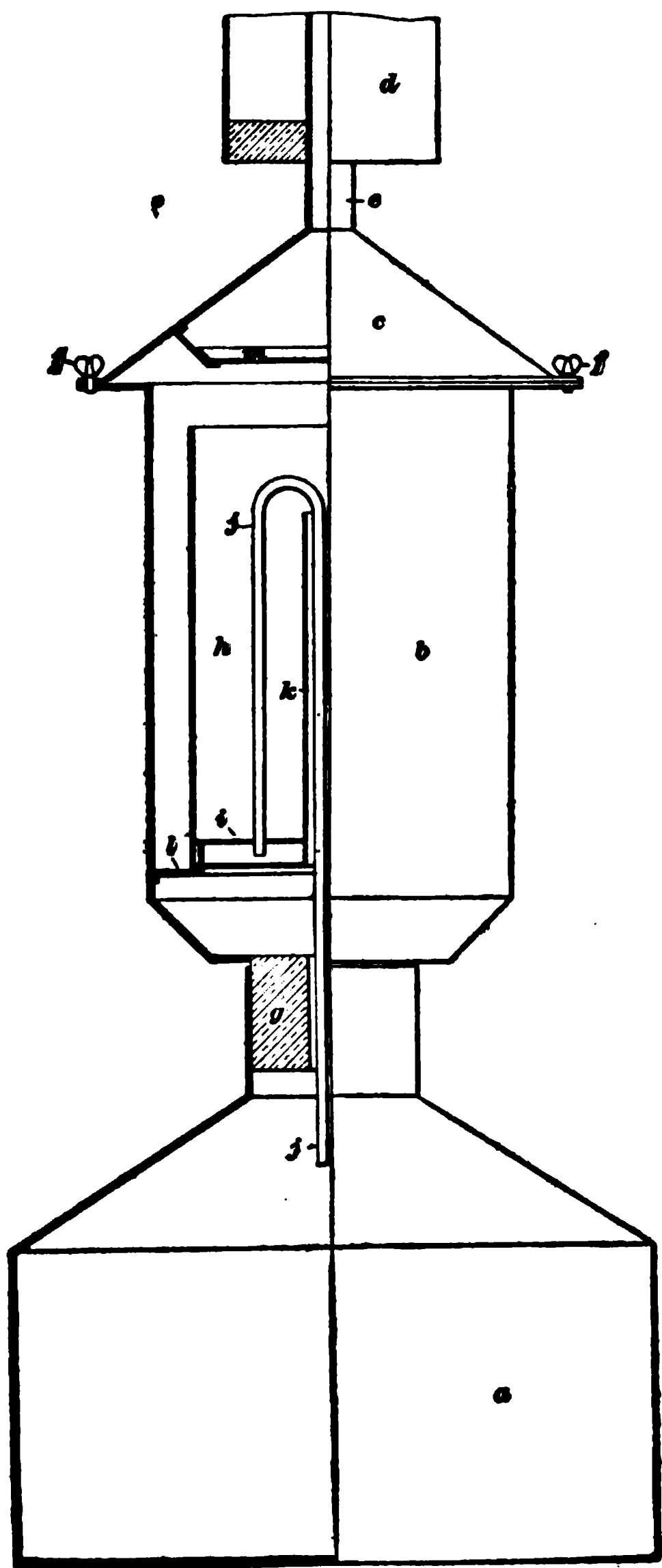


FIG. 19

into slots in the conical extractor cap *c*. A brass tube *e* runs from the apex of the extractor top through a condenser jacket *d*, having running-water connections. A movable brass cup *h*, which contains the material to be extracted, is in the upper can, resting on a perforated ring *g*; through the center of the can is an open tube *k*, forming a casing for a siphon *j* that carries the liquor from the bottom of the cup and, running through the perforated-brass plug, delivers it to the reservoir. A wire screen *i* prevents particles of bark mixing with the water that collects at the bottom of the can; a flanged screen *m* causes the condensed water to fall into the bark cup evenly over the entire surface. When heat is applied to the lower can containing the water, the steam rises through the perforated plug to the upper can, and finally through the brass tube surrounded by the water-jacket; here it is condensed and drops back into the bark cup; when this has filled to above the top of the siphon, the liquor passes through the siphon back to the reservoir, carrying back with it the soluble substances it has leached from the bark.

100. After cleaning the extractor, the bark is weighed into the cup and 1 liter of water is added in small portions, or as fast as it is siphoned off into the lower can. This is for the double purpose of wetting the bark well and to be sure that the siphon is working well. The top is then clamped tight, the water started through the condenser jacket, and the lamp under the extractor lighted. The flame must be regulated so that a constant simmering goes on; if the flame is too hot and the boiling too violent, the volume of condensed water returning will be too great and a violent bumping will be caused by the steam meeting it in the tube.

The extraction continues from 8 to 10 hours, according to the nature of the material. When completed, the extractor is cooled, unclamped, and the liquor in the reservoir can emptied into a glass cylinder; there will generally be from 800 to 900 cubic centimeters remaining of the original liter poured over the bark, the loss being due to leaks. The can

is rinsed well with cold water and the rinse water used to make up the liquor to the liter mark; after cooling, the liquor is ready for analysis.

101. Preliminary Remarks Regarding the Determinations to be Made.—Three determinations, the *total solids*, *soluble solids*, and *non-tannins*, are made in a tannin analysis, and from the results the *insolubles*, or *reds*, and the *tannins* are obtained. The difference between the total-solids figure and the soluble-solids figure gives us the amount of insolubles, or reds. By subtracting the non-tannin result from the soluble-solids results, we get the amount of tannins in the liquor. These terms being self-explanatory, it is not necessary to dwell on the definition of each, but a few words on some of their constituents will not be amiss.

The reds, or insolubles, have been defined under tanning materials; because of their greater or less solubility in warm liquor, it is necessary that the analysis should always be made when the liquor has a temperature of from 60° to 70° F.; if colder, the reds figure will be higher and the subsequent tannin result lowered; if warmer, the reds will decrease and the tannin content increase.

Under non-tannins we class all soluble substances in the liquor not absorbed by hide. They consist for the most part of sugary substances derived from the sap of the tree. In the same manner we include under tannins all soluble substances that will combine with hide, thus embracing all the forms of tannic acid.

102. Determination of Total Solids.—When the liquor, extracted and prepared as has been described, has cooled, the cylinder is shaken and then allowed to stand for $\frac{1}{2}$ hour; this is to allow small particles of bark to settle, so that they will not be counted as reds, or insolubles proper. At the expiration of $\frac{1}{2}$ hour remove with a pipette 100 cubic centimeters of the liquor, place in a weighed glass crystallizing dish, and evaporate to dryness on a water bath. When evaporated, place in a water oven and dry from 12 to

15 hours, cool in a desiccator, and weigh; from this weight the percentage of **total solids** is figured.

103. Determination of Soluble Solids.—For the **soluble-solids** determination, place 250 cubic centimeters of the liquor in a beaker, add about 8 grams of kaolin, stir, and filter through a double filter paper. The kaolin should be previously washed with hydrochloric acid, then with distilled water, and finally dried and pulverized; it is used as an aid in filtering, forming a thin film over the paper, thus insuring a clear filtrate. The paper and kaolin absorb tannin from the solution, so that the affinity of these for tannin must be satisfied by allowing 100 cubic centimeters of the liquor to pass through the paper before any of the filtrate is collected for evaporation. When 100 cubic centimeters have passed through the paper, replace the glass collecting the filtrate with a clean, dry glass and evaporate and dry 100 cubic centimeters of this second filtrate for the soluble-solids determination.

104. Hide Powder for the Determination of Non-Tannins.—To determine the **non-tannins**, hide powder is used. The best made comes from Europe and may be procured from any of the importers of chemicals. It is made by liming and depilating selected steer hides, neutralizing the lime remaining in the hide with hydrochloric acid, and removing the excess of acid by thorough washing. The hide is then carefully and slowly dried, and when dry is ground about as fine as sawdust. A good hide powder should absorb tannin quickly and contain *no* soluble hide substance. The hide powder from Europe possesses these qualities in a larger degree than the American product, but the best must be carefully washed before use. To prepare for use, proceed as follows: Soak the amount needed in about eight times its weight of water for an hour, then squeeze out as much water as possible, using a linen or muslin cloth, and add fresh water. Repeat this operation twice, making three washings in all, which is generally enough for a good grade of hide powder. After the third washing place the hide in

a stout cloth and squeeze as dry as possible in a hand press, such as is used for pressing fruit, etc. The hide powder is then ready for use in the non-tannin determination.

105. Chroming of Hide Powder.—Because of the difficulty encountered in procuring different lots of hide powder that will give concordant results, the practice of **chroming** the hide has lately been resorted to with excellent results in most of the large leather laboratories. The chroming operation makes the poorest hide powder equal to the best European product; it increases the absorption of the hide, and in some manner as yet not definitely understood gets rid of the soluble hide. It is thought that the chromium combines with soluble hide to form an insoluble compound, but whatever the action, hide powder chromed in a uniform manner will give clear non-tannins and concordant, reliable results.

The common method of chroming is to dissolve 3 grams of chrome alum for every 100 grams of hide powder to be chromed. The hide powder is washed with water once and fresh water run on it, then half of the chrome solution is poured in and thoroughly mixed. This should be done in the morning, and in the evening the remaining chrome solution poured on, mixed, and the hide powder allowed to stand over night. Four or five washings are given the next morning—enough to remove all trace of sulphates, and the hide powder is then ready for the final squeezing in the hand press.

106. Determination of Non-Tannins.—For the non-tannin determination, measure 200 cubic centimeters of the bark liquor into a strong glass tumbler, such as is used with a milk-shake machine, and after weighing 20 grams of the wet hide, to be placed in the steam oven and dried for moisture determination, weigh out from the press the amount of wet hide necessary for the determination, place in the liquor, breaking up the lumps with a glass rod, and then thoroughly shake. This may be done by hand, using a tin cap, such as is used for mixing drinks, or by a hand- or

power-driven milk-shake machine. Four minutes' shaking on a milk shaker is sufficient time for the hide powder to absorb every trace of tannin from the liquor. After shaking, add about 8 grams of kaolin and filter through double filter paper. When the filtrate is running clear, collect, and with a pipette remove 100 cubic centimeters to a crystallizing dish, evaporate, dry, and weigh as under the solids determination.

The amount of hide powder to be used depends on the strength of the bark liquor; in the case of a new bark, where a 25-gram sample has been extracted, 40 grams of the well-pressed hide is generally sufficient. For a liquor from spent tan bark, where an 80-gram sample has been extracted, 40 grams of hide powder is sufficient and 25 grams is often enough. Whether or not enough hide powder has been used can be judged by the appearance of the non-tannin filtrate; if clear and white or pale yellow, the tannin has been removed. If the filtrate has a red or brown tinge, it is generally safe to assume the presence of some tannin. A good test is to add to a doubtful filtrate a solution of gelatine; if tannin is present, a cloudy precipitate will at once form.

107. Calculation of Results of Bark Analysis.— Divide the weight of the total-solids residue by the number of grams of bark taken, and divide this result by the percentage of actual bark in the sample, found by subtracting from 100 the percentage of moisture, as determined in a 10-gram moisture sample. This result, when pointed off to correspond to the decimal part of the original liter, is the percentage of total solids. The percentage of total soluble solids is found in the same manner.

Because of the water added by the wet hide used in the non-tannin determination, a correction must be made. If the 20-gram sample of wet hide powder when dry weighs but 6 grams, there are 14 grams of water added to the liquor for every 20 grams of wet hide used. If 40 grams of wet hide is used, there are 28 grams of water added, and the volume then becomes 228 cubic centimeters instead of

the 200 cubic centimeters originally taken; 100 cubic centimeters of the thus diluted filtrate has been dried, and it accordingly represents only $\frac{100}{114}$ of the actual amount of non-tannins, so the weight must be multiplied in this case by 1.14. Therefore, to calculate the percentage of non-tannins, divide the non-tannin weight successively by the number of grams of bark taken and the percentage of dry bark, as under the solids calculations, and multiply this result by the hide-moisture correction.

ILLUSTRATION.—For the analysis, 25 grams of bark were taken and 40 grams of wet hide powder were used. The bark contained 12 per cent. of moisture and 88 per cent. of dry bark. A 20-gram sample of wet hide when dried weighed 7 grams. The 40 grams of hide used therefore contained 14 grams of dry hide and 26 grams, or cubic centimeters, of water, which latter was added to the original 200 cubic centimeters.

$$\frac{200 + 26}{200} = 1.13, \text{ or the hide-powder correction for moisture.}$$

Total-solids residue weighed.....	.4983 gram.
Total soluble-solids residue weighed.....	.4012 gram.
Non-tannin residue weighed.....	.1441 gram.

For the total-solids determination, 100 cubic centimeters of liquor were taken, or .1 of the liter. The calculation would be as follows:

$$\frac{.4983 \times 100 \times 10}{25 \times .88} = 22.65 \text{ per cent. of total solids.}$$

The total soluble solids are calculated in the same manner.

$$\frac{.4012 \times 100 \times 10}{25 \times .88} = 18.24 \text{ per cent. of total soluble solids.}$$

The total solids less the total soluble solids give the amount of insolubles,

$$22.65 - 18.24 = 4.41 \text{ per cent. of insolubles.}$$

For the determination of non-tannins, 100 cubic centimeters, or $\frac{1}{10}$ liter of liquor, were dried. The hide-powder correction was found to be 1.13. The calculation would be as follows:

$$\frac{.1441 \times 100 \times 10 \times 1.13}{25 \times .88} = 7.40 \text{ per cent. of non-tannins.}$$

The total soluble solids less the non-tannins give the amount of available tannin.

$$18.24 - 7.40 = 10.84 \text{ per cent. of available tannin.}$$

The complete analysis is as follows:

Total solids	{	Total soluble solids	{	Non tannins. 7.40	}	18.24	{	22.65%
				Tannins10.84				
				Reds, or insolubles.....		4.41		
Insoluble parts of bark (not removed by leaching).....								77.35%
Total.....								100.00%

ANALYSIS OF TAN LIQUOR

108. Determination of Specific Gravity and Total Solids.—Shake sample well and with a barkometer determine its specific gravity in degrees barkometer. For total solids, shake well and remove 10 cubic centimeters of liquor to a crystallizing dish, evaporate, dry, and weigh.

109. Determination of Total Soluble Solids.—For total soluble solids, mix 250 cubic centimeters of liquor with 8 grams of kaolin and filter through double-filter paper, allowing the liquor to filter for about 10 minutes to satisfy the affinity of the paper for tannin before collecting filtrate for evaporation. Evaporate 10 cubic centimeters of this second filtrate and dry and weigh.

110. Determination of Non-Tannins.—For non-tannin determination, it is best to dilute the liquor so that the resulting dilutions of the different liquors are comparatively weak. For a liquor of from 25° Bark. to 40° Bark., dilute 50 cubic centimeters to 500 cubic centimeters; from 15° Bark. to 25° Bark., dilute 100 cubic centimeters to 500 cubic centimeters; below 15° Bark., dilute 200 cubic centimeters to 500 cubic centimeters. Take 200 cubic centimeters of the diluted liquor, add hide powder, and shake 3 minutes; add kaolin, filter and evaporate, dry and weigh 100 cubic centimeters of the filtrate. The amount of wet hide to be used varies from 15 grams to 40 grams, according to the tannin strength of the liquor; a large excess will do no harm if the hide is well washed and the moisture correction carefully determined. For sulphuric-acid tanneries, it is desirable

to know the amount of free and combined sulphuric acid in the "liquor run to the creek."

111. Determination of Total Free Acids (Sulphuric and Organic).—If it is required to determine the free acid in a liquor, 100 cubic centimeters of the diluted liquor used for the non-tannin is measured into a beaker, animal charcoal stirred in and allowed to stand with frequent stirring for an hour or more. Then filter, take an aliquot part, dilute with distilled water, and titrate with $n/10$ sodium hydrate, using phenol-phthalein indicator.

112. Determination of Combined Sulphuric Acid. To determine the combined sulphuric acid, evaporate 100 cubic centimeters of the original liquor in a platinum dish and ignite the residue. Cool, add hydrochloric acid and hot water, filter off the insoluble matter, and determine the sulphuric acid in the filtrate with barium chloride, as explained in *Quantitative Analysis*.

113. Determination of Total Sulphuric Acid.—To determine the total acid, take 50 cubic centimeters of the original liquor, add distilled water and a little hydrochloric acid, bring to a boil, and precipitate the sulphuric acid direct with barium chloride. The difference between the percentages of total acid and combined acid will be the free sulphuric acid in the liquor.

114. Calculation of Results of Tan-Liquor Analysis. The total solids and total soluble-solids weights will give direct results when pointed off to percentage basis. On the non-tannins, if the dilution has been 50 cubic centimeters to 500 cubic centimeters, the result will also be direct. If dilution has been 100 cubic centimeters to 500 cubic centimeters, the weight must obviously be divided by two; if 200 cubic centimeters to 500 cubic centimeters, by four. The moisture correction must in all cases be applied to non-tannin weights.

ANALYSIS OF TANNING EXTRACT

115. General Remarks. — The exact conditions to which an extract analysis must conform in order that it may be said to have been made by the "Official Method," given later, are set forth annually in the report of the Association of Official Agricultural Chemists, the Tannin Branch of which association comprises the leading leather chemists in America.

As carried out now, the method is to dissolve such amounts of extract as will give approximately 8 grams of total solids to the 100 cubic centimeters in 900 cubic centimeters of water at a temperature of 80° C., allow to stand over night, make up to the 1,000-cubic-centimeter mark in the morning and proceed with the analysis. About 20 grams of a liquid extract and 10 grams of a solid extract are taken and made up to the liter.

116. Determination of Total Solids.—For total solids, shake well, evaporate 100 cubic centimeters to dryness and weigh. As a check on the total-solids determination, a moisture determination is made by weighing from 2 to 3 grams of the extract into a weighed crystallizing dish, cover with cold distilled water, and dry and weigh the same as in the other determinations.

117. Determination of Total Soluble Solids.—For total soluble solids, add about 8 grams of kaolin to 150 cubic centimeters of the dissolved extract, stir well, and filter through double Schleicher and Schull's No. 590 filter paper, allowing the entire 150 cubic centimeters to pass through the filter. When this has filtered through, throw out the filtrate, pour carefully fresh extract solution on the filter, and evaporate, dry, and weigh 100 cubic centimeters of this second filtrate. Evaporation during filtration is guarded against by inserting the stem of the filtering funnel in the neck of a 500-cubic-centimeter flask and collecting the filtrate in this receptacle.

118. Determination of Non-Tannins.—For non-tannins, shake 200 cubic centimeters of the solution with 50 grams of hide powder for 5 minutes, then strain through cloth, using hand pressure, add kaolin to the strained liquid, mix and filter through double filter paper. Evaporate, dry, and weigh 100 cubic centimeters for non-tannins. The operation of squeezing through a cloth is merely for the purpose of hastening the subsequent filtering. With 50 grams of hide powder in 200 cubic centimeters of water, filtering would be a tedious operation; so the liquid is squeezed out and then filtered with kaolin.

119. Calculation of Results of Tanning-Extract Analysis.—The results are calculated by dividing the different weights by the amount of extract taken and pointing off for the percentage basis, not forgetting the hide-moisture correction on the non-tannin weight.

OFFICIAL METHODS FOR ANALYSIS OF TANNING MATERIALS

120. The following methods for the analysis of tanning materials, issued in Circular 8 of the Bureau of Chemistry of the United States Department of Agriculture, were adopted at the Eighteenth Convention of the Association of Official Agricultural Chemists.

1. *Preparation of Sample.*—Barks, woods, leaves, dry extracts, and similar tanning materials should be ground to such a degree of fineness that they can be thoroughly extracted. Fluid extracts must be heated to 50° C., well shaken, and allowed to cool to room temperature.

2. *Quantity of Material.*—In the case of bark and similar material, use such quantity as will give from .35 to .45 gram of tannins per 100 cubic centimeters of solution, extract in Soxhlet or similar apparatus at steam heat for non-starchy materials. For canaigre and substances containing like

amounts of starch, use temperature of 50° to 55° C. until near complete extraction, finishing the operation at steam heat. In the case of extract, weigh such quantity as will give from .35 to .45 gram of tannins per 100 cubic centimeters of solution, dissolve in 900 cubic centimeters of water at 80°, let stand 12 hours, and make up to 1,000 cubic centimeters.

3. *Moisture*.—(a) Place 2 grams, if it be an extract, in a flat-bottom dish not less than 6 centimeters in diameter, add 25 cubic centimeters of water, warm slowly until dissolved, continue evaporation, and dry.

(b) All dryings called for, after evaporation to dryness on water or other baths, shall be done by one of the following methods, the soluble solids and non-tannins being dried under similar and, so far as possible, identical conditions:

I. For 8 hours at the temperature of boiling water in a steam bath.

II. For 6 hours at 100° C. in an air bath.

III. To constant weight in vacuo at 70° C.

4. *Total Solids*.—Shake the solution, and without filtering, immediately measure out 100 cubic centimeters with a pipette, evaporate in a weighed dish, and dry to constant weight at the temperature of boiling water. Dishes should be flat-bottomed and not less than 6 centimeters in diameter.

5. *Soluble Solids*.—Double-pleated filter paper (S. and S., No. 590, 15 centimeters) shall be used. To 2 grams of kaolin add 75 cubic centimeters of the tanning solution, stir, let stand 15 minutes, and decant as much as possible. Add 75 cubic centimeters more of the solution, pour on filter, keep filter full, reject the first 150 cubic centimeters of filtrate, evaporate the next 100 cubic centimeters, and dry. Evaporation during filtration must be guarded against.

6. *Non-Tannins*.—Prepare 20 grams of hide powder by digesting 24 hours with 500 cubic centimeters of water and

adding .6 gram of chrome alum in solution, this solution to be added as follows: One-half at the beginning and the other half at least 6 hours before the end of the digestion. Wash by squeezing through linen, continue the washing until the wash water does not give a precipitate with barium chloride. Squeeze thoroughly by hand and remove as much water as possible by means of a press, weigh the pressed hide, and take approximately one-fourth of it for moisture determination. Weigh this fourth carefully and dry to constant weight. Weigh the remaining three-fourths carefully and add them to 200 cubic centimeters of the original solution; shake for 10 minutes and squeeze the tanned hide through linen. Collect this filtrate, add 5 grams of kaolin, free from soluble salts, stir well, and filter through folded filter, returning the first 25 cubic centimeters. Evaporate 100 cubic centimeters of the clear filtrate. The weight of this residue must be corrected for the dilution caused by the water contained in the pressed-hide powder. The shaking must be done in some form of mechanical shaker. The simple machine used by druggists, and known as the "milk shake," is recommended.

Provisional Method.—To 14 grams of dry, chromed hide powder in a shaker glass add 200 cubic centimeters of the tanning solution, let stand for 2 hours, stirring frequently, shake 15 minutes, throw on funnel with a cotton plug in the stem, let drain, tamp down the hide powder in the funnel, return the filtrate until clear, and evaporate 100 cubic centimeters.

7. *Tannins.*—The amount of these is shown by the difference between the soluble solids and the corrected non-tannins.

8. *Testing Non-Tannin Filtrate.*—(a) *For Tannin.*—Test a small portion of the clear non-tannin filtrate with a few drops of a 1-per-cent. solution of Nelson's gelatine. A cloudiness indicates the presence of tannin, in which case repeat the process described under 6, using 25 instead of 20 grams of hide powder.

(b) *For Soluble Hide.*—To a small portion of the clear non-tannin filtrate add a few drops of the filtered tannin solution. A cloudiness indicates the presence of soluble hide, in which case repeat the process described under 6, giving the hide powder a more thorough washing.

The temperature of solutions shall be between 16° and 20° when measured or filtered. All dryings should be made in flat-bottom dishes of at least 6 centimeters in diameter. S. and S. No. 590, 15-centimeter filter paper should be used in all filtrations for the removal of insolubles.

MANUFACTURE OF SOAP

(PART 1)

INTRODUCTION

1. Definition of Soap.—By the term **soap**, in its strict acceptation, is meant the compound of an alkali, either sodium or potassium, with the higher fatty acids, especially with oleic, palmitic, and stearic acids. The insoluble compound of a fatty acid with a heavy metal is, however, technically called a soap. The chemist is familiar with the lead soap or lead plaster of the pharmacy, with alumina soap used as a thickener of lubricating oils, and with iron and chromium soaps used in dyeing and in the color printing of textiles.

As commonly known, soap is, according to its quality and the use for which it is intended, a mechanical mixture of the compound described above, with varying proportions of water, with soluble alkali compounds of the rosin acids, with sal soda Na_2CO_3 , $10 H_2O$, with sodium silicate or soluble glass, or with other inert, deterative, or odoriferous agents, incorporated for the purpose of cheapening the product, improving its appearance, increasing the deterative action, or overcoming the natural odor with an agreeable perfume. Therefore, commercial soap is a mixture of pure soap with

a diluent, as water; with body-imparting substances, as talc, starch, or a petroleum residue; with deterative agents in aqueous solution, as sodium carbonate, borax, or sodium silicate. These additions may all be present in a single soap, but the nature and amount of the additions present depend on the character of the soap itself and the purpose for which it is intended.

2. Classes of Soap.—We may define three general classes of soap, viz., *industrial*, *household*, and *toilet*. In the first class are included so-called textile soaps used in the process of manufacture of cotton and woolen goods. In the second class are included those soaps that are consumed in various qualities and conditions by the laundry trade; and in the third class may, conveniently, be included shaving and medicinal soaps.

In the discussion of the subject, we shall be guided more by the process of manufacture than by the use intended for the product.

GENERAL OUTLINE OF THE MANUFACTURE OF A SETTLED SOAP

3. The following outline gives a general idea of the various operations performed in the manufacture of a settled laundry or domestic soap. The relation of the different operations to one another is here made clear and the following detailed descriptions of the different operations and processes rendered more intelligible.

Source of Fatty Acids.—In the definition of a soap in Art. 1, it was stated that a soap is a salt formed by the union of an organic acid of the higher fatty series with a metal, usually an alkali metal. The acid portion of soaps is derived from various animal and vegetable fats and oils, which are compounds of the above-mentioned organic acids with glycerine.

Soap Stocks.—Any fat or oil used in the manufacture of soap is called a **soap stock**, and may be either of animal

or vegetable origin. The various kinds and grades of soap stock used will be fully discussed farther on.

Saponification.—This is the operation by which oils and fats are combined with the alkali metals sodium or potassium, to form soaps. Technically, this term has a wider meaning, and applies to any operation that brings about a separation of an organic acid from an organic base.

When an animal or vegetable fat is boiled with a caustic alkali, a double decomposition takes place. The products are the alkali salts of the organic acids of the fats and glycerine.

Graining and Settling.—After the saponification is complete, the soap is **grained** or brought into a somewhat granular condition by the addition of certain salts or alkalis in whose solutions the soap is practically insoluble. When in this granular state the soap separates from the impure liquor, or *niger*, which may be drained off.

Crutching.—The soap, after the above treatment, is introduced into a machine called a **crutcher**, where it is crutched or stirred until it is thoroughly mixed. Here, fillers, perfumes, etc. are introduced and incorporated into the soap.

Framing.—From the crutcher the soap is transferred to a rectangular tank, called a **frame**, of proper dimensions, in which it is allowed to stand until of a suitable consistency for cutting into slabs and bars.

Slabbing.—The mass of solidified soap from the frame is first cut horizontally into slabs. This operation is called **slabbing** and is performed on a machine called a **slabber**. These slabs are in turn cut, on somewhat similar machines, into blocks of suitable dimensions for making finished bars.

Drying, Pressing, Etc.—The rough blocks are now dried superficially until a thin skin is formed over them. They are then pressed into finished bars in dies and wrapped and packed for shipment.

RAW MATERIALS OF SOAP MANUFACTURE

ANIMAL SOAP STOCK

4. General Remarks.—Animal soap stock occurs on the market in a variety of grades, depending on its origin and method of preparation. It constitutes the chief fatty material used by the soap maker. According to the part of the animal from which it is obtained and the method of rendering, it may be classified as **tallow**, **bone stock**, and **grease**. These classes of animal soap stock may again occur in various grades. In determining the quality, buyers, as a rule, depend on the simple tests of color, odor, and grain, supplemented by the titer, or hardness, test, described later. In addition, the percentage of moisture, melting point, and percentage of free fatty acids are determined. The last determination is of especial importance when the stock is considered with relation to its yield of glycerine.

5. Tallow.—Tallow varies in its composition according to the part of the animal from which it is obtained and the nature of the food used in fattening the animal. Corn-fed cattle produce the firmest fat. The fat of mast-fed cattle is not so firm as that obtained from animals fattened on oil cake. Tallow consists of about 60 per cent. of stearin, 34 per cent. of olein, and 6 per cent. of palmitin.

6. The percentage of free fatty acids is a reliable index not only of the quality of the stock used by the renderer but a telltale upon the care which he has used.

The following conditions conducive to the formation of a high percentage of free fatty acids in the various grades of tallow may be enumerated:

1. Allowing the raw material to stand before rendering, especially in warm weather. After the death of the animal, decomposition immediately sets in, and the first step in the decomposition of tallow is the separation of the fatty anhydrides from the glycerol.

2. An excessively high temperature in rendering tends to increase the percentage of free fatty acids. The presence of water in the tissues and the influence of the high temperature in the rendering tank tend to the hydrolysis of the glyceride.

3. Rendering in closed vessels increases the percentage of free fatty acids. It will be clear that there prevail here conditions that are purposely introduced and maintained in the process for the saponification of glycerides with steam under pressure in the manufacture of fatty acids and glycerine.

7. **Bone Stock.**—This material is intermediate in quality between tallow and grease and is obtained from fresh bones by rendering in a closed vessel under pressure.

8. **Grease.**—This is a general term applied to all fatty material of animal origin inferior in quality to the two general grades already mentioned. It is obtained from sources not generally contributing to the grades of tallow and bone stock, and frequently may be superior in quality. Dead animals, both cattle and swine, are an abundant source. It occurs in a great variety of grades, determined as a rule by its origin, whether it be from dead animals, kitchen refuse, recovered garbage, or butchers' offal. From animals dead in transit, white, brown, and yellow grease is obtained, which is equal to tallow in soap-making qualities.

9. The relative quality of these three general grades of animal soap stock, as shown by the free-fatty-acid determination and the influence of the season, is indicated in the following table:

PERCENTAGE OF FREE FATTY ACIDS.

Soap Stock	Summer	Winter
Tallow.....	7.80	5.85
Bone stock.....	10.68	6.64
Grease.....	18.03	7.49

The significance of the free-fatty-acid determination will be fully shown when the various fats and oils are discussed with reference to their content and yield of glycerine and the changes incident to saponification.

MANUFACTURE OF ANIMAL SOAP STOCK

10. The various grades of tallow are extracted from the fat-enclosing tissue that surrounds the intestines, muscles, and other organs of the animal. The feet yield specifically neatsfoot oil. The process of rendering consists essentially in the separation of the fatty matter from the enclosing animal tissue. Tallow was originally obtained by simply boiling the finely divided parts of the animal containing the fat in water and skimming from the surface of the water the fat thus disengaged from the membrane. Fleishy portions of the animal previously finely chopped and rendered in the above manner yield the kettle-rendered tallow used for edible purposes. Tallow, other than that of packing-house origin, is usually distinguished as country or city rendered. The former, being from fresher stock, yields a better quality of tallow.

11. Steam Rendering. — Steam-rendered tallow is obtained by subjecting the rough fat to a steam pressure of 30 to 60 pounds per square inch in a closed vessel, such as is shown in Fig. 1. The top of the tank is on the floor level and is provided with a safety valve, a manhole *a* for introducing the rough fat commonly conveyed in barrows, and a discharge pipe for the exit of fumes. Near the middle of the tank are two draw-off cocks (not shown in the figure) for the removal of the liquid tallow. Water is admitted through a valve near the bottom. At the bottom of the tank is either a large gate valve *b* or a tightly fitting door for the discharge of the "soup." The *soup*, or *soup liquor*, as it is called in the packing house, consists of soft bones, solid residuum, and water highly impregnated with albuminous matter. The liquid portion is run off and evaporated to a sirupy consistency under a partial vacuum and is afterwards

mixed with the solid matter freed from an excess of liquor by compression in a fertilizer press. This material constitutes the *tankage* fertilizer of the packing house.

After charging, these tanks, usually arranged in a series, are tightly closed. Water is run in, if desired, and steam is turned on. The period of working varies from 4 to 10 hours, according to the size of the tanks, the quantity of material introduced, and the steam pressure. At the expiration of the rendering period, the fat is discharged by means of the draw-off cocks. When the last portions of fat are below the level of the draw-off cocks, water is admitted into the tank from below, whereby the fat floating upon the surface of the water is completely discharged. The remaining contents of the tank is then dropped through the discharge gate at the bottom.

12. Chemical Rendering.—Processes have been devised for the separation of fat from the animal membrane by chemical means, but they are not employed in practice. Practically all the animal stock used in the manufacture of soap is prepared by the steam-rendering process.

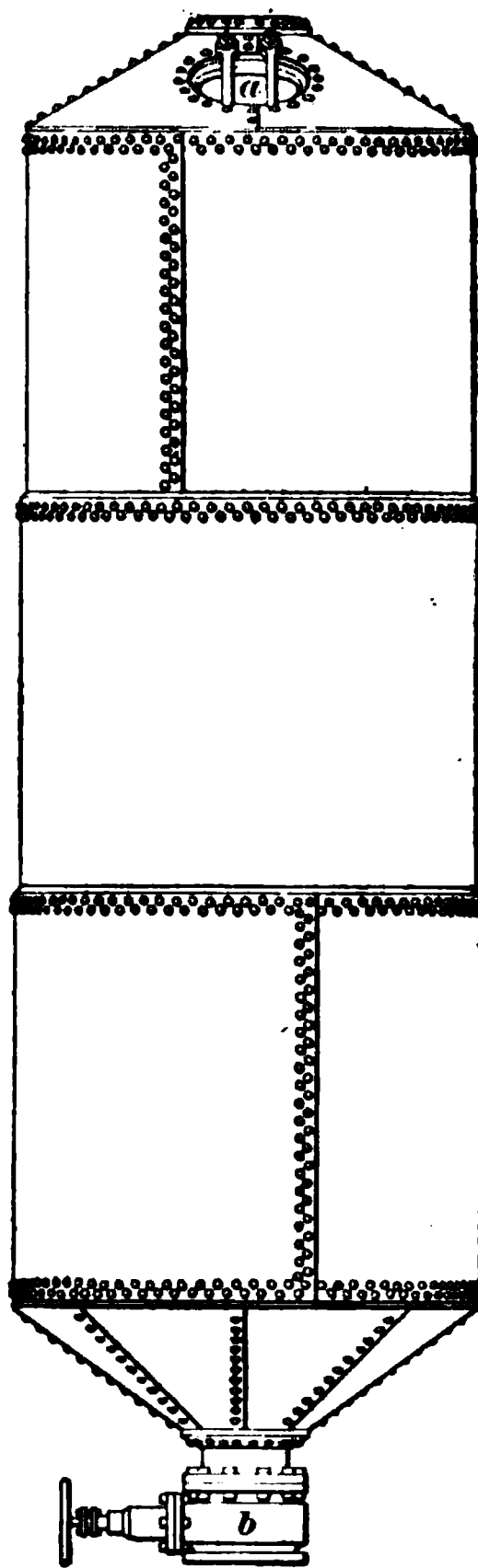


FIG. 1

VEGETABLE SOAP STOCK

13. General Remarks.—Any oil may be employed for the manufacture of soap, its applicability for this purpose being determined by its character, cost, and the use intended for the soap made from it. Corn oil and the various nut

oils—peanut oil, for example—cannot compete with cottonseed oil because of their greater initial cost. Castor oil is used but little, its very limited consumption being confined to the manufacture of transparent soaps. Linseed oil is used extensively in Europe in the manufacture of soft soap. In this country, soft soap finds but limited sale as a detergent. The soft soaps used in the cotton and woolen industries are made from a non-drying glyceride base. In Table I is given the average percentage yield of oils of vegetable origin. The percentage yield of oil from seeds, fruits, etc. is dependent on a variety of conditions, chief among which are the character of the soil, the weather, and the degree of ripeness, and are subject to extreme variations. These conditions influence, as well, the specific gravity of one and the same oil. Moreover, according to the age, mode of preparation, etc. of the oil, the variations in specific gravity may be as great as the difference between the density of one oil and that of another which serves as an adulterant.

TABLE I

YIELD OF OIL FROM VARIOUS SEEDS, NUTS, ETC.

Seed	Percentage Yield of Oil
Castor (Indian).....	51-53
Castor (American).....	46-49
Coconut	40-45
Colza, or rape.....	33-43
Cottonseed.....	24-26
Linseed.....	38-40
Maize, or corn.....	6-10
Olive (pericarp).....	40-60
Palm (pericarp).....	65-72
Palm kernel.....	45-50
Peanut.....	43-45
Sesame.....	50-57

14. Cottonseed Oil.—Cottonseed oil is the chief product of the cottonseed, which constitutes about two-thirds of the weight of the unginned cotton grown in the Southern States. In recent years it has attained great importance as a soap stock, although its use in this connection is small in comparison with its consumption for edible purposes. Crude cottonseed oil is a thick, reddish-brown to black oil of extremely varying quality, depending on the season and the care exercised in the collection and storage of the seed. The crude oil contains from 10 to 15 pounds of coloring matter per ton of seed. This coloring matter has been isolated, but is of little practical value. The loss in refining the crude oil amounts to from 5 to 7 per cent. This is an extremely varying factor, depending on the percentage of free fatty acids and the proportion of coloring and albuminous matter present, or, briefly, on the character of the seed. The yield of oil depends on the soil, season, and skill of manufacture, and ranges from 35 to 42 gallons of crude oil per ton of seed.

15. Refining Crude Cottonseed Oil.—In the refining process, the first step is to separate as quickly and as thoroughly as possible the foreign matter from the expressed crude oil. The residue obtained on settling or filtration is known as *settlings* or *foots*. The precipitate or residue obtained in the refining process is sometimes termed "soap stock," but is known in the cottonseed trade as *cottonseed foots*, under which name it is sold. ("Foots" is a loose term, at best, and "foots" and "soap stock" are frequently used indiscriminately.) The crude oil freed from settlings is now pumped into the first-treatment tank, where it is agitated, under increased temperature, with caustic-soda lye of strength and proportion adapted to the quality of oil undergoing treatment. When sufficient time has elapsed for the complete absorption of the alkali, the agitation is ceased and the contents of the kettle is allowed to remain at rest, during which time the soap, formed by the combination of the free fatty acids with the caustic alkali, contaminated with the organic impurities of the oil, settles to the bottom.

The supernatant oil thus partially clarified is then transferred to the second-treatment tank, where it undergoes a bleaching process with fuller's earth. The proportion of fuller's earth used varies with the quality of the oil and may run from 2 to 5 per cent., and often a second treatment with fuller's earth after filtration is required.

The oil after agitation with fuller's earth, under increased temperature, is passed through the filter press. The filtrate constitutes the prime summer yellow cottonseed oil used in soap making. The various grades of edible and industrial cottonseed oils are obtained from this product by subsequent treatments with fuller's earth and filtration. The winter cottonseed oil used in miners' lamps requires limpidity at a low temperature, which condition is obtained by chilling the oil to a temperature below 0° C., whereby the so-called cottonseed oil "stearin," which is really palmitin, separates.

16. Grades of Cottonseed Oil.—The Texas Cottonseed Crushers' Association requirements for the various grades of cottonseed oil compel a uniformity of quality which is absent in the soap stock of animal origin. The summer yellow oil employed in soap manufacture is usually examined for color, moisture, free fatty acids, and titer, the last test being of chief importance in determining comparative value for soap-makers' use.

17. Crude Cottonseed Oil.—The following are rules of the Texas Cottonseed Crushers' Association relating to cottonseed oil.

Measurement.—A tank (tank car) of cottonseed oil shall be 125 barrels. A barrel of oil, if sold loose, shall contain 50 gallons. A gallon of oil shall weigh $7\frac{1}{2}$ pounds avoirdupois.

Classification.—Crude cottonseed oil shall be classed and graded as follows:

Choice crude must be made from sound decorticated seed, must be sweet in flavor and odor, light in color, free from water and settlings, and test not over 1 per cent. of free fatty

acids. It shall produce, when properly refined, choice summer yellow oil at a loss in weight not exceeding 6 per cent. for Texas oil, and at a normal loss for oil from all other parts of the country.

Prime Crude.—Crude cottonseed oil to pass as *prime* must be made from sound decorticated seed, must be sweet in flavor and odor, free from water and settlings, and must produce prime summer yellow grade by the usual refining methods with a normal loss in weight, provided the oil shall not be rejected for a nominal amount of settlings; but reasonable reduction shall be made in value for all such settlings in excess of $\frac{1}{4}$ per cent.

Off Oil.—All oil neither choice nor prime shall be called *off oil*, and shall be sold by sample.

18. Refined Cottonseed Oil.—Refined cottonseed oil shall be classed and graded as follows, summer yellow oil only being considered:

Choice summer yellow must be sweet in flavor and odor, of light straw color, clear and brilliant in appearance, free from moisture, and must bleach to a choice white.

Prime summer yellow must be clear, sweet in flavor and odor, and of yellow color, not reddish, and free from water and settlings.

Off Oil.—This grade consists of all oils having any objectionable flavor or odor, or are of a reddish color.

19. Cottonseed-Oil Soap Stock.—The precipitate obtained from the crude oil freed from settlings, on treatment with caustic soda lye, consists of soap and free oil mixed with mucilaginous and coloring matter. This mixture undergoes further treatment with caustic alkali, whereby all the free oil is transformed into soap. The finished soap is grained and subjected to further brine and alkali washings in order to obtain a product as free from odor and as light in color as possible. Cottonseed-oil soap stock is, therefore, essentially a soap. After it has been suitably purified and on the final settling, it is run while hot and in a semi-fluid condition into barrels.

20. Requirements of the Texas Cottonseed Crushers' Association for Soap Stock.—All sales, unless otherwise agreed upon by buyer and seller, are on a basis of 50 per cent. of fatty acid, not to fall below 40 per cent. If containing less than 40 per cent. of fatty acid, soap stock shall not be considered merchantable. Delivery is to be made in iron-bound hardwood packages or tank cars. A tank car of soap stock shall be 50,000 pounds for contract purposes.

21. Coconut Oil.—Coconut oil is obtained from the fruit of the coco palm, the *Cocos nucifera* of the tropics. The oil is expressed from the pulp of the nut, which contains about 50 per cent. of oil. It occurs on the market in three general grades, viz., Cochin, Ceylon, and copra oil. The terms Cochin and Ceylon have reference more to the geographical origin of the fruit than to a definite gradation of quality. Some Ceylon oil is equal if not superior to the Cochin variety.

Cochin coconut oil is expressed from fruit grown in Cochin, China, and adjacent territory, and on the islands of the China Sea.

Malabar and Manila oil is of good quality. Mauritius and the Fiji Islands also produce considerable. The coco palm is a native of Cuba, and at one time oil crushing was a thriving industry on that island.

Ceylon coconut oil is a product of the island of Ceylon and of the adjacent regions. The different grades of coconut oil arise from the varying skill and nature of the apparatus employed in its recovery, some of the latter of which are very crude. Where improved milling machinery has been introduced, coconut oil of uniform and superior quality is obtained; the greater part of the oil, however, is extracted by the most primitive methods. The oil arrives at the factory in immense casks or pipes containing upwards of 2,500 pounds.

Copra coconut oil is made from the dried pulp or meat of the coconut, called copra. It yields, on steaming and pressing, the lowest grade of coconut oil. The best oil of

this grade is obtained from the ripest and most quickly pressed copra. Owing to its high percentage of the lower fatty acids and to its greater exposure, the copra oil is more liable to rancidity than is oil of Ceylon and Cochin grades. The Ceylon and Cochin oils are expressed in the districts near the shipping ports, while copra is produced almost entirely in territory deficient in shipping facilities. Great quantities of copra are shipped to Marseilles, France, where the oil is expressed. In Sydney, New South Wales, copra crushing is a thriving industry.

22. Coconut oil as a soap stock possesses distinct and peculiar qualities that define it sharply from all other animal and vegetable oils. In this connection, it may be well to note that there is no sharp distinction between a fat and an oil. The term fat is usually applied to those glycerides that are solid at ordinary temperatures and is usually of animal origin; while an oil may be of either animal or vegetable origin and is liquid at ordinary temperatures.

23. Palm-Kernel Oil.—Palm-kernel oil in its chemical composition and behavior towards saponifying agents is closely allied to coconut oil. It finds but limited application in this country as a soap stock. It is expressed from the kernel of the palm nut, the fleshy envelope of which is the source of palm oil. The oil is white and has an agreeable odor and taste, but on aging it becomes rancid. Oudemans gives the following as the approximate composition:

GLYCERIDE	PERCENTAGE
Olein.....	26.6
Stearin }	33.0
Palmitin }	
Myristin }	
Laurin }	44.4
Caprin }	
Caprylin }	
Caproin }	

24. Palm Oil.—Palm oil is expressed from the fleshy part of the fruit of the palm tree, the *Elæis Guineensis* and

the *Elæis melanococca*, native along the west coast of Africa. According to its source and mode of expression, it is of butterlike to tallowy consistency, varying in color from orange to dark red, and possesses a characteristic violet-like odor. The oil is bleached on exposure to the air; chemical bleaching removes the odor as well. The color and odor persist in the soap made from it. Its chief constituents are free palmitic acid, palmitin, and olein. The behavior of palm oil in the soap kettle resembles closely that of tallow. The high percentage of free fatty acids arises from the primitive methods employed in its extraction. The chief industrial chemical test applied to palm oil is the determination of the titer.

25. Corn Oil.—Corn oil is expressed from the germ obtained as a by-product in the manufacture of starch and glucose from maize. It is closely allied to cottonseed oil in its soap-making properties. It is usually of a bright yellow color, but may be bleached with fuller's earth to a whiteness, as is done with certain edible grades of cottonseed oil. The lower price of cottonseed oil militates against its general use as a soap stock. It finds its chief use as a cheapener of paint oils.

In the manufacture of the oil, the corn is first steeped in water, whereby the hull is loosened from the grain, which becomes swollen and tough from the absorption of water. The steeped kernel is then passed between rollers to separate the envelope and the starch from the softened germ, which remains whole and of the size of a grain of rice. The crushed corn is then transferred to tanks filled with water, in which the crushed grain sinks, leaving the germs to float upon the surface. These are removed, cooked, and compressed for the oil.

26. Olive Oil.—Olive oil is expressed from the pulp or fleshy portion of the fruit of the olive tree, the *Olca Europæa*, native in the Mediterranean countries, where it has been employed from the earliest time as the fatty base of *Castile soap*. The use of olive oil for this purpose has of late years been largely displaced by the cheaper cottonseed oil, large

quantities of the latter being exported from the United States to Marseilles for this purpose. Olive oil is used chiefly as an edible oil and inferior grades only are used in the soap kettle. Olive oil finds very limited use in this country as a soap stock. It contains about 72 per cent. of olein and 28 per cent. of palmitin. It behaves like cottonseed oil towards saponifying agents.

In the manufacture of olive oil, about 10 bushels of olives are crushed at a time in an edge-stone mill. The pasty mass resulting, consisting of pulp and stones, is then placed in filter cloths and subjected to hydraulic pressure. The first pressing of the olives yields the finest oil. After the first pressing, which yields the virgin oil—as it is sometimes called—the press cake is ground up with water in the edge-stone mill and the paste then subjected to a second pressing. The second pressing yields an oil of inferior quality. The press cake after the second pressing yet contains a considerable quantity of oil, which, when recovered, constitutes the olive-oil foots of commerce.

27. Olive-Oil Foots.—The oil remaining in the press cake after the second pressing may be extracted in either of two ways, but when recovered it is of such inferior quality as to be suitable only for industrial purposes. It is fairly fluid, of dark green to black color, and of disagreeable odor. Its inferiority results from the contamination by the green coloring matter and pulp of the fruit. To recover the remaining oil, the press cake is first well ground with the addition of hot water. The dark pasty mass is then transferred to a tank and agitated with water until the broken olive stones drop, free from pulp, to the bottom of the tank. The oily residuum floating on the surface of the water is then transferred to another tank, where the free oil is removed. The pulp residuum is again pressed and yields some oil. Olive-oil foots obtained in this manner is said to be “washed.”

The press cake may also be extracted with a volatile solvent, carbon disulphide being commonly used. The broken press cake is agitated with the solvent, after which

the solution thus obtained is transferred to a covered tank. Here the solvent is distilled off and collected for subsequent use, leaving the recovered oil behind. This oil is black and retains the odor characteristic of the solvent. It is inferior to "washed" foots. This process, however, possesses the advantage of complete extraction of the oil.

Soap made from olive-oil foots retains the green color of the recovered oil; the color, however, gradually bleaches out on exposure.

28. Red Oil.—Red oil is a by-product obtained in the manufacture of candles and consists almost wholly of oleic acid. It derives its name from the color that the oil acquires upon aging, but chiefly due to contact with the iron. It occurs in two grades—viz., "saponified" and "distilled" red oil.

Saponified red oil is obtained by either acid, lime, or aqueous saponification under pressure, in specially constructed tanks called autoclaves or digesters. The mixed fatty acids resulting from saponification are resolved roughly into stearic and oleic acids by pressure through filter cloths. The stearic acid yet retained in the liquid portion is separated by chilling, whereby the commercial oleic acid, or red oil, is obtained.

Distilled red oil is obtained by distillation of the mixed fatty acids with steam. Through the process of fractional condensation, fatty acids of different melting points are separated. Red oil varies in its composition according to the method employed in its manufacture. The following are analyses of two samples of commercial oleic acid :

INGREDIENTS	SAMPLE A	SAMPLE B
Color.....	Pale	Brown
Oleic acid.....	93.06%	87.70%
Oil.....	6.04%	9.41%
Hydrocarbons90%	2.89%
Total.....	100.00%	100.00%
Specific gravity.....	.897	.904
Turbid at.....	42° F.	38° F.

Red oil is a valuable soap stock. It admits of saponification with soda ash, as will be fully explained elsewhere.

29. Manufacture of Saponified Red Oil.—This, as stated, may be by three different methods of saponification—viz., lime saponification, acid saponification, and aqueous saponification.

30. Lime Saponification.—The digester or autoclave is a stoutly built tank, usually of copper, although some are made of iron, from 3 to 5 feet in diameter and from 18 to 25 feet in height. The digester may be set up either horizontally or vertically. It is covered with an asbestos jacket, to retain the heat. It is provided with a safety valve, a cock for the removal of samples, and a pressure gauge. At one end are placed pipes for the introduction of the tallow and lime and for the discharge of the contents of the digester after saponification and for the introduction of live steam.

The tallow is previously purified, if required, by boiling in weak brine and allowing the impurities to subside. The brine wash is then run off and the tallow maintained in a fluid state by means of a closed steam coil. The quantity of unslaked lime commonly used for saponification is from 2 to 4 per cent. of the weight of the tallow. Both the tallow and lime tanks are on an elevation above the digester, and after the lime has been thoroughly mixed with water and the tallow melted, they are allowed to run by gravity into the digester. The charging of the digester may be hastened by creating a partial vacuum in it by the condensation of steam, introduced previously to running in the charge.

After the charge has been added, steam is turned on and maintained at a pressure of from 8 to 10 atmospheres for a period of from 4 to 10 hours, or until the saponification is complete; 8.7 per cent. of lime is theoretically required, but under the conditions maintained in the digester, from 2 to 4 per cent. has been found in practice to be sufficient. Samples are removed from time to time and tested for

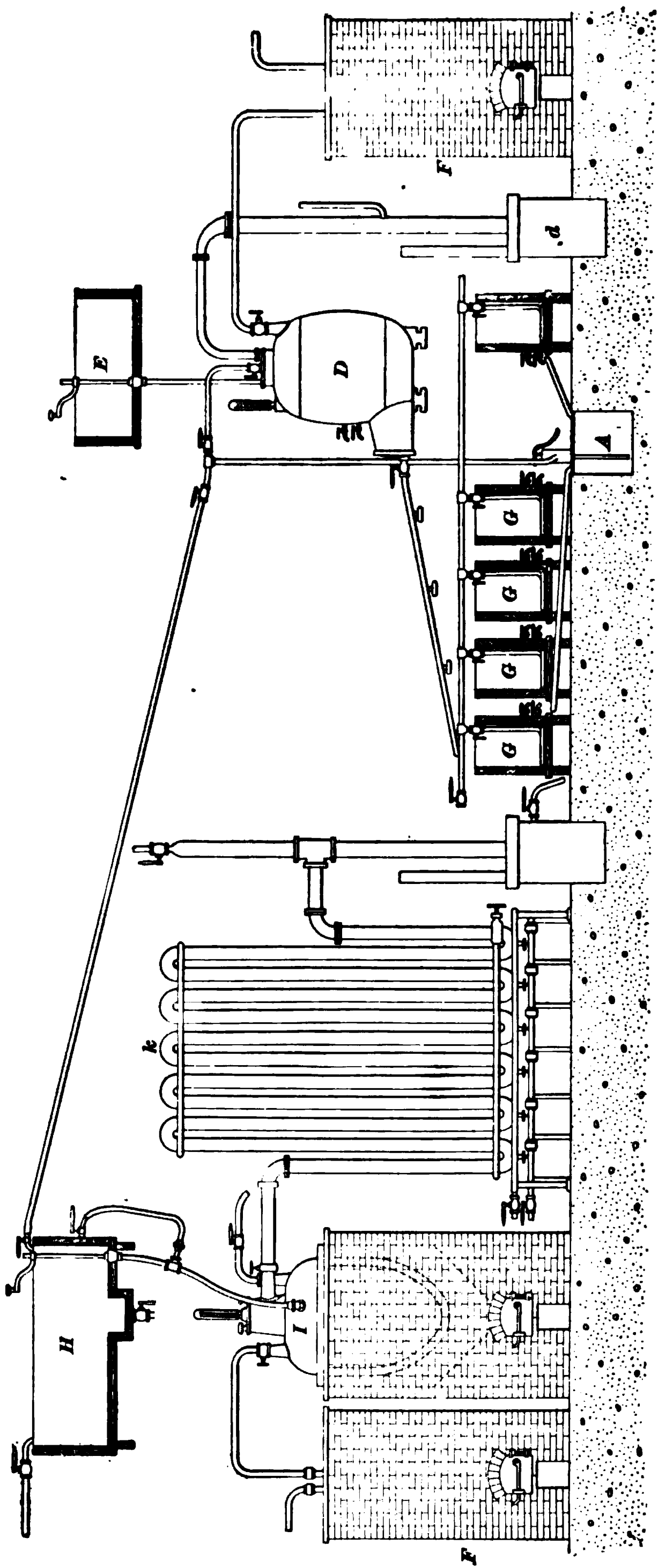


FIG. 2

unsaponified matter. On completion of saponification, the contents of the digester is blown into wooden tanks or vats placed above the digester.

The mass resolves itself into two layers, the supernatant lime "rock" consisting of lime soap and fatty acids, and the "sweet" water, in which is dissolved the glycerine liberated from the stock. The glycerine solution is now allowed to flow by gravity to the glycerine plant. The lime rock is again boiled up with water and live steam in order to remove the last traces of glycerine. This wash liquor is removed as before.

The decomposition of the lime rock is effected by adding slowly, under constant agitation, the calculated quantity of dilute sulphuric acid. The fatty acids of the lime soap are thereby set free. The mass at once resolves itself into two layers: the supernatant layer, consisting of the total fatty acids, and the water containing the calcium sulphate in solution and as a precipitate. The acid liquor and precipitate are now discharged into the sewer and the fatty acids washed free from all traces of sulphuric acid.

31. Acid Saponification.—In Fig. 2 is shown a type of apparatus of English manufacture employed in the acid-saponification process for the manufacture of fatty acids for candle stock.

The raw material, whether it be tallow, palm oil, recovered grease, or any fatty body containing stearin, is first melted in the tank marked *A* and is then transferred to the storage tanks *G*. These are wooden and lead-lined and provided with acid-resisting steam coils. In these storage tanks the fat undergoes a preliminary purification, as already briefly described. The mass is then transferred to the acidifier marked *D*. It is here treated with from 4 to 12 per cent. of concentrated sulphuric acid, which is introduced by gravity from tank *E*, immediately above. In the acidifier *D*, the mixture is subjected to the action of superheated steam furnished by the superheater *F*. To condense the acid vapors evolved during the reaction, a jet condenser provides a

slight vacuum in the acidifier whereby the vapors are drawn over and discharged into the reservoir *d*.

After the acidification is complete the material is discharged into the storage tanks *G*, where the acid liquor is removed and the fatty acids washed free from all traces of acid.

32. Aqueous Saponification.—Many establishments manufacturing candle stock employ neither acid nor lime in the hydrolysis of the glyceride, but saponify their tallow, bone fat, or other stock merely in the presence of water and under 150 pounds steam pressure. Saponification by means of water is the simplest and most convenient of the three processes described, inasmuch as the lime and acid treatments are made unnecessary. The process otherwise is carried on in precisely the same manner as described in the lime-saponification method.

33. Distillation of the Fatty Acids.—The fatty acids, whether obtained by the lime, acid, or aqueous saponification, are subjected to the same process of distillation. When made of pure and fresh raw material, the resulting fatty acids are of sufficiently good color to press immediately. The dark-colored fatty acids before pressing are subjected to distillation with superheated steam. Referring to Fig. 2, the fatty acids are pumped to the charging tank *H* situated on an elevation above the still *I*, and into which it may flow by gravity. The still, generally of copper or copper lined, is of variable capacity. A still of ordinary size will accommodate 16,000 to 18,000 pounds at a “run,” during which the volume of material in the still is kept constant by the addition of fatty acids from the charging tank above. As shown in Fig. 2, the still is encased in brickwork and heated externally by fire and within by superheated steam passed through copper coils from the superheater *F*. The products of distillation are condensed in the vertical cooling tubes *k*, connecting at the bottom with coils immersed in warm water. The condensed fatty acids are here melted to admit of their easy removal. The distillate is commonly

collected in three fractions—viz., the first-run oil, comprising three-fourths of the total charge; the second-run oil, which is returned to the charging tank and redistilled with the next run; and the final portion, called from its color *green oil*. The residue in the still is known as *candle tar*.

34. Pressing the Fatty Acids.—The fatty acids, whether having been subjected to a previous distillation or used directly from the autoclave, are transferred to square, shallow pans or trays supported on shelves in the granulating room. These trays are of enameled iron and are so placed that each tray, beginning at the end, is at a higher elevation and slightly over the succeeding one. It is thus possible to fill every pan in the series by running the melted fatty acids in the topmost, which, when full, overflows into the succeeding one, and so on until all are filled. Were the fatty acids cooled rapidly, the crystals of the fatty acids of different melting points would be so closely interlocked that the oleic acid could not be readily separated from the stearic. By allowing the mixture of fatty acids to cool slowly for a period of from 2 to 3 days in the granulating room, at a temperature of about 80° F., the stearic acid crystallizes in a menstruum of the liquid oleic acid. There is thus obtained at the expiration of this stage of the process a cake of stearic acid colored brown by the oleic acid.

The pan is now inverted on a woolen or camel's-hair cloth, upon which the cake falls. When the required number of cloths have been filled, they are transferred to the hydraulic press. Each cloth and its contents is separated by an iron plate. Pressure is applied very gradually at first, whereupon the crude oleic acid or red oil is expressed. It is conducted to storage tanks, from which it is run to the department in which it is used or barreled for shipment.

The first pressing separates about 50 per cent. of the oleic acid present and is done cold.

The cakes thus obtained are subjected to a second pressing between hollow iron plates heated with steam. The hot pressing is effected in a horizontal hydraulic press. The

stearic acid now obtained is of snowy-white appearance and very hard and brittle. It melts at 52° to 55° C. and is ready for the candle manufacturer.

35. Comparison of the Processes for the Manufacture of Fatty Acids.—The yield of solid fatty acids from tallow by the lime saponification is from 44 to 48 per cent. Aqueous saponification admits of a slightly higher yield, viz., 50 per cent. from the same raw material; acid saponification yields upwards of 55 per cent. of fatty acids. It is found in practice that distilled stock yields a press cake of lower melting point than that obtained from fatty acids used directly from the autoclave. This fact, taken together with the greater yield of the acid-saponification process, with which process distillation is used, may be explained by the polymerizing action of the sulphuric acid upon the oleic acid in the still, whereby a body is formed of sufficient firmness to resist being expelled from the press cake with the crude oleic acid, and therefore remains to lower the melting point and to add to the yield.

With lime saponification, practically all the glycerine, upwards of 10 per cent., is obtained; with the acid saponification, not more than 3 per cent. is recovered.

If it is desired to recover as much glycerine as is possible, in addition to the solid fatty acids, the lime saponification is preferable.

If fatty acids are the sole desideratum, the distillation process is better.

36. Rosin.—Rosin is the solid residue left on the distillation of crude turpentine. The rosin or crude turpentine exudes from the pine tree, chiefly the "long-leaf" yellow pine of the Southeastern Atlantic and Gulf States. This rosin is of whitish color and of semi-solid consistency. A deep incision is made in the trunk of the tree about 1 foot from the ground. Each incision or "box" holds about a quart of the crude rosin, the number of boxes depending on the size of the tree. As the season advances the flow of rosin is increased by removing the bark and wood to the

depth of an inch above the box. At regular intervals, the exuded rosin is collected and distilled. The still (see Fig. 8) is of copper and varies in capacity from 10 to 50 barrels of crude turpentine. It is mounted on brickwork with a furnace underneath. After the still has been charged, some water is run in and the contents of the still heated. The fire is increased gradually until the contents of the still has reached the boiling point, where the temperature is maintained until practically all the volatile

FIG. 8

matter has been distilled over. As the distillation proceeds, water is added from time to time to replace that lost by evaporation and to prevent darkening of the product by incipient burning. The distillate, separated from the water that has distilled over with it, constitutes the oil of turpentine of commerce. The hot liquid residuum in the still is now discharged through a valve near the bottom and is strained through sieves of increasing mesh up to No. 80, usually three in number, and is finally run into barrels. A charge of 12 barrels of crude turpentine, weighing upwards of 4,500 pounds, will yield on distillation about 3,600 pounds of rosin and about 900 pounds of oil of turpentine.

37. The barrels contain upwards of 500 pounds of rosin and are marketed on the basis of 280 pounds to the barrel. There are about fifteen grades of rosin, varying in color from "water white," which is clear and almost colorless, to the lowest grade, commercially known as "C. A.," which is black. The various qualities arise from the length of time during which the crude rosin is collected from a single "camp."

The first year's run furnishes the best grades, while with each succeeding year a more inferior grade is

obtained. The following grades are used in the soap industry:

W. W., or Water White.

W. G., or Window Glass.

N., or Extra Pale.

M., or Pale.

K., or Low Pale.

Rosin is graded by sample, a $\frac{1}{8}$ -inch cube being cut from the head of each barrel. Uniformity of size is important, as the thickness of the cube determines the shade of color, and therefore the value. Those who buy and sell rosin are provided with sample cubes representing the quality of the standard grades. The determination of the quality of a shipment is, therefore, the simple matter of comparing the sample cubes with those of the standards.

38. Rosin is acid in its composition, consisting of the anhydrides of the so-called rosin acids. Like red oil, it admits of saponification with soda ash. The alkaline salts of the rosin acids, while not constituting a true soap, possess marked detergent properties and are a valuable ingredient of household soap. The English were the first manufacturers of rosined soaps; Americans soon copied them and greatly improved the product.

As rosin decreases in quality from the brighter to the darker grades, the percentage of unsaponifiable matter increases, as is indicated by the following determinations of the unsaponifiable matter:

Marks	Percentage Unsaponifiable Matter
W. W.	3.07
W. G.	3.88
N.	4.08
M.	6.34
K.	6.62

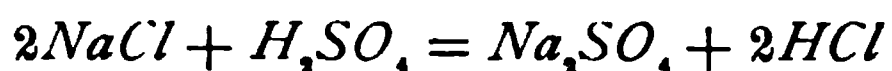
The coloring matter present in rosin is the cause of the high color of the waste soap lye withdrawn after the rosin change in the manufacture of settled rosin soap.

ALKALIS AND THEIR MANUFACTURE

39. Soda Ash.—All the commercial sodium compounds start from common salt as a raw material. Before the development of the Le Blanc process, potash was used exclusively as the saponifying agent in the primitive soap manufacture of those days. This was obtained by burning seaweeds and lixiviating the ashes. The solution thus obtained was then causticized with quicklime. During the embargoes of the French Revolution, the supply of potash was cut off from France. As the result of a prize offered by the French Government for a practical method for the manufacture of soda ash from common salt, the Le Blanc process was given to the world. So perfectly was it outlined in its original specifications that it has undergone no change in its essential principles in its entire history.

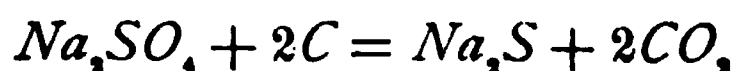
40. The Le Blanc Process for the Manufacture of Sodium Carbonate.—The manufacture of soda ash, or sodium carbonate Na_2CO_3 , from common salt by the Le Blanc process is carried on practically in three stages, as expressed in the following reactions:

I. Common salt, or sodium chloride, is converted into sodium sulphate by treatment with sulphuric acid.

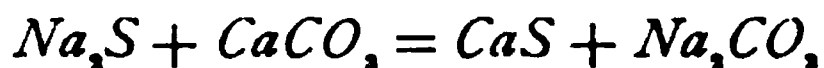


The hydrochloric-acid gas is absorbed by passing it through towers down which water is allowed to trickle. The aqueous solution of the gas thus obtained constitutes the muriatic acid of commerce.

II. The sodium sulphate obtained by the preceding reaction is mixed with coal and fused in specially constructed furnaces. The coal reduces the sulphate to sulphide.



III. The sodium sulphide is now heated with carbonate of lime, which then reacts to form calcium sulphide and sodium carbonate.



The product of this reaction is crude soda or black ash. The fused mass is allowed to cool and is then broken into fragments and lixiviated with water. The sodium carbonate in solution is allowed to crystallize out as $Na_2CO_3 \cdot 10H_2O$. The water of crystallization is expelled by heat, leaving the sodium carbonate or soda ash of commerce.

41. The Le Blanc process, a creation of French ingenuity, enjoyed its greatest development on English soil, and for half a century was one of the chief mainstays of England's industrial supremacy. The practical employment of the process involved certain technical and many unsanitary disadvantages, whose baneful effects proved a constant encouragement for the development of a simpler and more hygienic process. The mechanical difficulties surrounding the production of alkali by what is now known as the *ammonia process* met their first practical solution at the hands of Ernest and Alfred Solvay, and in the past thirty years there have been established in every civilized country works operating the process originally covered by the Solvay patents. The first ammonia soda works were established in Belgium in 1863. Later, immense works were established in England by Brunner, Mond & Co., whose name is indissolubly associated with the development of the process in Great Britain.

The Le Blanc process has received the most thorough scrutiny of the best scientific minds, and the efforts made to enable it to survive in the face of the more economical production of a competing process have resulted in the most complete economy of operation. As a remunerative producer of alkali, the ammonia process early displaced it, with the result that the process is dependent for its profits entirely on its chlorine products. As yet no economical production of bleaching powder from the calcium chloride waste of the

ammonia soda process has been wrought out on an extensive commercial scale, and as these two rival chemical processes stand today, the Le Blanc has command of the chlorine industry, while the scepter of remunerative alkali production has passed to the ammonia process.

42. Solvay, or Ammonia Soda, Process.—The Solvay process for the production of soda ash from common salt, is based on the precipitation of sodium bicarbonate in an ammoniacal solution of common salt by means of carbonic-acid gas. Practically all of the soda ash now produced is obtained by this process.

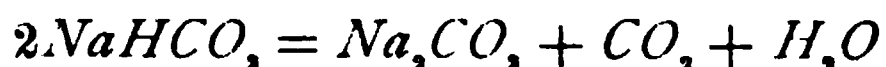
In the practical operation of the Solvay process the employment of labor is reduced to a minimum. The materials employed in the direct manufacturing processes are either in a gaseous state or in aqueous solution. The fundamental chemical reaction of the process is as follows:



The salt is dissolved in water to form a very pure and concentrated brine. It is then saturated with ammonia gas. The liquor thus formed is introduced under pressure into the "carbonating" tower at a distance about one-third from the top. The carbonating tower is upwards of 65 feet in height and is made up of segments of about 3.5 feet high and 6 feet in diameter. The carbonic-acid gas is forced into the tower through the bottom segment and is forced to ascend in bubbles by means of a perforated plate covering a hole in the bottom of each segment. The reaction indicated by the above formula takes place in the carbonating tower. The success of the ammonia-soda process depends on the insolubility of the sodium bicarbonate in a cold ammoniacal solution of common salt. The heat produced by the chemical reaction is taken up by cold water circulating in cooling pipes placed in each segment of the carbonating tower.

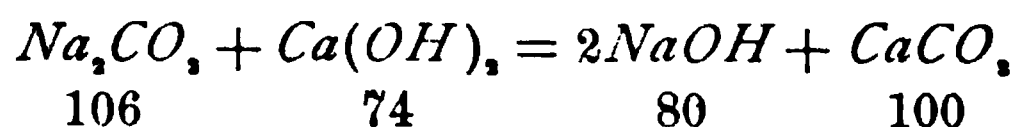
43. As the reaction proceeds, the precipitated sodium bicarbonate accumulates in the bottom of the tower, where it is withdrawn from time to time as a thick, milky liquid,

containing ammonium chloride and sodium chloride in solution and the sodium bicarbonate in suspension. The solid bicarbonate is separated from the chlorides of ammonium and sodium in solution by means of centrifugal machines and is then washed with water to remove traces of these impurities. The bicarbonate is then calcined to form a normal carbonate in accordance with the following reaction:

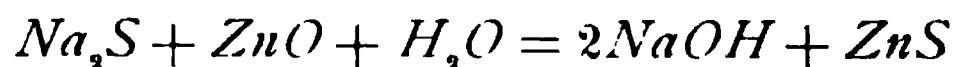


The Solvay soda ash thus obtained has a specific gravity of only .8, owing to its extremely finely divided condition. To increase the ease of handling it and to bring it up to the density of Le Blanc soda ash, viz., 1.2, it is made more compact by subjecting it to a second ignition. It is then ground and packed in bags and casks for shipment. Soda ash made by this process is much purer than the Le Blanc soda ash. It contains only traces of salt and bicarbonate and is free from the sulphide, sulphate, and hydrate of sodium.

44. Caustic-Soda Manufacture.—The manufacture of caustic soda is based on the following reaction:



According to this reaction, 100 pounds of sodium carbonate will yield on causticization 75.4 pounds of sodium hydrate. In the manufacture of caustic soda from the soda ash of the Le Blanc process, the lime is added to the solution obtained by lixiviating the black ash. The solution must not have a density in excess of 13° Baumé, or a reversion of the calcium carbonate will ensue. The impure solution of sodium carbonate is therefore diluted, if necessary, and the "milk of lime" added. Under heating and agitation with air, the reaction represented by the above formula takes place. Zinc oxide is added to reduce the sodium sulphide; thus,



The addition of sodium nitrate, together with the air used for agitation, suffices to oxidize any thiosulphate to the

normal sulphate. The solution is then allowed to settle and the supernatant solution of caustic soda is removed and evaporated until a density of 33.5° Baumé is attained, at which point salts present as impurities crystallize out. The solution is then transferred to a heavy cast-iron pan and the evaporation is continued over fire until all water is expelled and the caustic soda remains as a fused mass.

A sample representing the contents of the kettle is tested for total alkali. If found to be below 60 per cent. quality, the contents is worked up to that test. If the test indicates the quality to be above 70 per cent., the product may be worked into the highest grade made or into that grade testing most closely to the contents of the kettle. The reduction of quality to the grade of caustic desired is effected by the addition of salt. The fused caustic soda is then run directly into sheet-iron drums and sealed to prevent exposure to the atmosphere. Owing to the greater purity of the solutions, it is possible to obtain purer caustic soda from soda ash by the Solvay process easier than it is from Le Blanc soda ash. With Solvay soda ash it is not necessary to remove the impurities always characteristic of the commercial products of the Le Blanc process.

45. Although the production of caustic lye from soda ash by the soap maker may be effected at a nominal cost, the use of the solid caustic, aside from other considerations peculiar to each works that may determine the installation of a causticizing plant, possesses superior advantages as regards cleanliness and convenience of working. The commercial production of solid caustic dates from 1854, at which time improvements introduced in its manufacture by William Gossage, in England, resulted in its more general use, although in a very impure state, by soap manufacturers and paper makers. Artificial alkali was first used in the manufacture of soap in 1823. It was manufactured in England by James Muspratt, according to the Le Blanc process. The Lancashire soap boilers were loath to accept this new and purer article, and it was only after Muspratt

had distributed gratis scores of tons of Le Blanc soda that they became convinced of the superior economy of artificial over natural soda. The use of the sheet-iron drums was introduced by Thompson in 1857, this innovation being a most welcome improvement over cooling the liquid caustic on iron slabs and subsequently breaking it into pieces and packing in barrels for shipment.

46. Causticization of Soda Ash in the Soap Factory. The causticizing plant consists essentially of a converting kettle, in which the chemical reaction indicated in Art. 44 is carried out, and a filter for the separation of the *lime mud*. The causticizing plant is shown in its essential characteristics in Fig. 4. It is desired to causticize the soda

FIG. 4

ash as completely as possible, and to this end the lime should be free from impurities. In causticizing a ton of soda ash, lime containing 2 per cent. of calcium sulphate will convert 42 pounds of a ton of soda ash into the less valuable product, sodium sulphate.

The solution of the soda ash in water is effected in the converting kettle *a* with the aid of live steam introduced through the pipe *b*, and the required amount of lime is shoveled in. The contents of the kettle is thoroughly agitated and boiled up with live steam, after which it is allowed to settle. The supernatant liquor is then pumped off through the swing-joint pipe *c* shown in the illustration. The lime precipitate, or mud, is boiled up with water and dropped upon the filter *D*, where it is drained and washed. In some instances this lime waste is recovered to be used as whiting, which is utilized in the manufacture of putty and in the paper industry as a make-weight, or filler.

47. As shown by experiments carried out by Lunge, the results of which are given below, the causticization is most complete with dilute solutions of soda ash.

TABLE II
COMPLETENESS OF CAUSTICIZATION OF SOLUTIONS OF
SODA ASH OF VARIOUS STRENGTHS (LUNGE)

Per Cent. Na_2CO_3 in Liquor	Sp. Gr. Before Causticizing	Per Cent. Na_2CO_3 Made Caustic by Treatment	
		<i>Test No. 1</i>	<i>Test No. 2</i>
2	1.022 at 15° C.	99.4	99.3
5	1.052 at 15° C.	99.0	99.2
10	1.107 at 15° C.	97.2	97.4
12	1.127 at 15° C.	96.8	96.2
14	1.150 at 15° C.	94.5	95.4
16	1.169 at 30° C.	93.7	94.0
20	1.215 at 30° C.	90.7	91.0

It is claimed for the apparatus shown in Fig. 4 that caustic-soda solution of 14.5° Baumé, equivalent to 600 pounds of 77 per cent. caustic, can be obtained from 800 pounds of 58-per-cent. soda ash and 650 pounds of lime. As a solution of caustic soda of this density is too dilute when soap

is boiled on open steam, evaporation of the weak caustic lye to the required density is resorted to when the facilities of the factory permit.

48. Electrolytic Production of Caustic Soda.—It has long been known as a scientific fact that when a current of electricity is passed through a solution of sodium chloride that the same is decomposed into its positive and negative ions, appearing, respectively, at the negative and positive poles, or, as they are called, the cathode and anode.

When a solution of sodium chloride is thus electrolyzed, chlorine will appear at the anode and sodium at the cathode. Chlorine, being gaseous, will either pass into solution or be discharged into the atmosphere; sodium, being a very active element, immediately decomposes the water surrounding the cathode, forming sodium hydrate and liberating hydrogen. Many varieties of electrolytic cells have been designed, with varying degrees of success, to serve as a commercial producer of caustic soda and chlorine. The chief mechanical difficulty in the production of a satisfactory electrolytic cell resides in the character of the diaphragm, which serves to separate the products liberated from the positive and negative poles. Chlorine must not be allowed to diffuse through the brine, for secondary reactions are set up. The problem is to find a satisfactory medium that will prevent the diffusion of the liberated products and at the same time offer no resistance to the passage of the electric current.

49. Castner Electrolytic Process for the Production of Caustic Soda and Chlorine.—In the Castner cell mercury is used to separate the chlorine liberated at the positive pole, or anode, and the sodium liberated at the negative pole, or cathode. The cell is essentially a box made of slate slabs and divided into three compartments marked *e*, *f*, *g*, in Fig. 5. Compartments *e* and *g* contain the salt solution and the carbon anodes *a*. Compartment *f* contains the caustic-soda solution and the iron cathode *c*. Sufficient mercury covers the bottom of the cell to alloy with the sodium as it is liberated. By means of the cam *b* the cell is

subjected to a slight oscillation upon the pivot d , whereby the sodium-mercury amalgam is carried into compartment f , where the amalgam acts as the anode to the iron cathode c , the sodium being set free to combine with the water in compartment f . The electrolysis of the brine takes place in compartments e and g . The sodium immediately alloys with

FIG. 5

the mercury, which serves as a vehicle to carry the alkali into compartment f , where the formation of caustic soda takes place. A regulated supply of water is run into the middle compartment to combine with the sodium, which is allowed to flow out as a dilute caustic-soda solution in corresponding volume. The chlorine gas is removed by exhaustion from compartments e and g .

The Castner process is employed industrially at Niagara Falls for the production of caustic soda and bleaching powder, and appears to be one of the most successful devices of its kind that has yet been developed.

50. Comparison of the Methods of Alkali Manufacture.—The Le Blanc process for the manufacture of commercial alkali products is able to survive in the face of the cheaper Solvay process with its absence of waste products only in virtue of its production of hydrochloric-acid gas as a by-product.

So long as no cheaper method for the manufacture of muriatic acid is developed, the Le Blanc process will always

constitute a considerable industry. In other respects, the Solvay process possesses every advantage.

The several electrolytic processes that have taken practical shape during the past few years have assumed a productive importance that will increase with each succeeding year. The problem of their commercial success is being gradually brought to a satisfactory solution, and what influence they may exert on the firmly established chemical processes is being watched with no little interest. As a producer of alkali, it is not likely that the ammonia process will be assailed. The older chemical process, which has so long enjoyed a monopoly of the manufacture of bleaching powder, has most to fear from the growth of the electrolytic methods.

To sum up the essential differences between the chemical and electrolytic methods for the production of sodium compounds, it may be stated that the electrolytic process is direct, clean, economical of labor, and free from worthless by-products. On the other hand, its units of plant are small, troublesome, expensive, and rapidly deteriorate in use. The chemical process provides a large output with comparatively few units of plant, of rather simple construction, and although of large size, the repairs, though heavy, are not numerous nor have they to be applied to a vast number of small pieces of apparatus. To the disadvantage of the chemical process, it may be said that arduous and fairly skilled labor is required for its operation and a number of by-products, invariably troublesome and of little or no value, are produced.

51. Grading of Soda Ash.—The system of grading soda ash and caustic soda is based on the molecular composition of these bodies. The quotations of the various grades in terms respectively of 48 per cent. alkali and 60 per cent. caustic is a vestigial characteristic of the early Le Blanc days and an evidence of the highest grades of those products they were then mechanically able to produce. The molecular weight of sodium carbonate Na_2CO_3 is 106,

composed of 62 parts by weight, or 58.49 per cent. of Na_2O , the remainder being CO_2 . A soda ash that contains 58.49 per cent. of Na_2O is therefore chemically pure, this percentage being equivalent to 100 per cent. Na_2CO_3 . A 58-per-cent. alkali should contain 58 per cent. of Na_2O or its equivalent, 99.16 per cent. Na_2CO_3 ; likewise, a 48-per-cent. alkali should contain 48 per cent. Na_2O or its equivalent, 82 per cent. Na_2CO_3 . The reduction of any grade of soda to that of 48 per cent. is effected by admixture with common salt. Below are presented for comparison two fairly representative analyses of these two standard grades of soda ash.

Grade	% Na_2CO_3	% $NaCl$	% Na_2SO_4	% $NaOH$	$F_2O_3, Al_2O_3,$ SiO_2	$CaCO_3,$ $MgCO_3$	H_2O
48%	60.64	28.34	4.85	1.29	1.12	Traces	4.26
58%	98.72	.54	.20		.10	.17	.26

52. Grading of Caustic Soda.—Caustic soda occurs on the market in a variety of grades and is sold on the basis of 60 per cent. of Na_2O . Caustic soda as a product of the alkali industry did not appear until 30 years after the industry was established in Great Britain, and the expression of its quality in the same terms as that of soda ash might be expected. The molecular weight of caustic soda is 40; to arrive at sodium oxide Na_2O , as an expression of the customary unit, two molecules, with a total molecular weight of 80, are used. In $2NaOH$ there are 62 parts, or 77.5 per cent. Na_2O , the remainder being H_2O . Therefore, a chemically pure caustic soda contains 77.5 per cent. Na_2O , or its equivalent, 100 per cent. $NaOH$. In the following table is given for comparison the percentage of the essential ingredient corresponding to, but never present in, the various grades of caustic commonly found in the market:

Grade	% $NaOH$
60	77.42
70	90.32
72	92.90
74	95.48
76	98.06
77	99.35
77.5	100.00

Sodium chloride, sodium carbonate, and sodium sulphate, in varying proportions, constitute chiefly the remainder of the ingredients. With the present system of grading based on the chemical determination of the total alkali, the Na_2O of the Na_2CO_3 is estimated with the Na_2O in terms of which the caustic soda, or $NaOH$, is expressed. With this method of expressing the quality of the caustic, the soap maker has just cause for complaint in that a variable percentage of a worthless ingredient is included in the total percentage of the essential ingredient present. The following is an industrial analysis of a sample of commercial caustic purporting to be of 74-per-cent. quality:

Total alkali Na_2O	74.18%
Total alkali, present as $NaOH$	69.88%
Caustic alkali $NaOH$	90.18
Combined alkali Na_2CO_3	7.35
Sodium chloride $NaCl$	2.02

This analysis indicates the sample to be of substantially 70-per-cent. quality, the difference between the sodium hydrate actually present and that claimed, viz., 4.30 per cent. Na_2O , is due to the 7.35 per cent. Na_2CO_3 , this being estimated as its equivalent, 4.30 per cent. Na_2O , in the total sodium oxide. As more or less carbonate is invariably present in all commercial caustic, especially in the lower grades, the system of including it in the expression of the quality of this product is open to severe criticism. Quotations of quality are thus confessedly a misrepresentation. The only rational method is the expression of the Na_2O as free caustic, or preferably units of $NaOH$.

This would be an absolute index of the value of the caustic as a saponifying agent, and not as by the method in vogue, an uncertain approximation of the same. English degrees indicate the strength of the ash or caustic in terms of Na_2O , but owing to either a wilful or accidental error in atomic weights, English analyses indicate a greater percentage of Na_2O than is actually present. This error has become so firmly established by tradition that modern ideas have been

unable as yet to eliminate it. In Germany and Russia, the strength is expressed in terms of sodium carbonate. This system is perfectly rational when applied to soda ash, but is inconsistent when applied to caustic. The expression of the value of commercial caustic soda in terms of an impurity, which, in so far as the soap industry is concerned, is positively worthless as a saponifying agent for neutral glycerides, is certainly not conducive to clear ideas on the subject.

53. The superior advantages and economy of high-grade caustic need no argument. It is true of this product that the best within certain limits is the cheapest. There is presented in Table III the price per 100 pounds of sodium hydrate as it occurs in the customary grades of caustic, assuming that no carbonate is estimated as caustic.

TABLE III

PRICES OF SODIUM HYDRATE OF VARIOUS GRADES

Grade	Price per 100 Lb.	%NaOH Present	NaOH per 100 Lb.
60%	\$1.65	77.42	\$2.131
70%	1.50 for 60%	90.32	1.937
74%	1.60 for 60%	95.48	2.066
76%	1.70 for 60%	98.06	2.196

The anomalous variation in prices quoted arises from the slight differences in cost of production of the lower grades, combined, for those grades, with the proportionally greater cost of packages, transportation, etc.

The total charges contingent upon marketing a 60-per-cent. caustic are the same as those of a 70-per-cent., although the former contains considerably less of the essential ingredient; also, the cost of production of a 70-per-cent. caustic is but little more than that of a 60-per-cent. The increased cost of production of the higher grades, viz., 74 per cent. and

76 per cent., makes necessary a higher price, which is less than it would be if cost of marketing were correspondingly increased.

54. The Preparation of Caustic-Soda Lye.—The solid caustic soda is delivered at the works in the familiar sheet-iron drums, containing usually about 750 pounds.

The dissolving of the solid caustic is a simple operation. The method employed in effecting solution, arrangement of the tanks, and system of transferring the lye, vary with each establishment.

The work of solution is facilitated by inserting a false bottom, or cage, in the melting tank to support the solid caustic previously broken into lumps. With this arrangement a natural circulation of liquor of different densities is set up, with the result that weak liquor continually rises to the surface, while the saturated liquor, in virtue of its greater density, subsides. This eliminates the necessity of mechanical agitation when the solid caustic is allowed to rest on the bottom of the tank.

55. In the preparation of caustic lyes of different densities from various grades of caustic, the influence of the impurities, chiefly sodium chloride, sodium carbonate, and sodium sulphate, is to reduce the active value of the solution for the specific gravity indicated. This reduction in saponifying power is least for the highest grades and greatest for the lowest, as a natural result of the increased percentage of these impurities present. There is arranged in Table IV the percentage of sodium hydrate present in lyes of different densities, made from the usual grades of caustics, corresponding to the densities of lye made from chemically pure caustic. It is assumed that the total alkali is present entirely as caustic, which never actually occurs. The figures stated, for reasons previously given, are generally slightly higher than would be found in practice. However, the table possesses some value as a basis of comparison, and for many technical purposes the figures are sufficiently accurate:

TABLE IV

PERCENTAGE OF SODIUM HYDRATE NaOH IN LYES OF
DIFFERENT DENSITIES MADE FROM CAUSTIC
OF VARIOUS GRADES

Specific Gravity	Degrees Baumé	77½% % NaOH	76% % NaOH	74% % NaOH
1.075	10	6.55	6.42	6.25
1.091	12	8.00	7.84	7.63
1.116	15	10.06	9.86	9.60
1.142	18	12.64	12.40	12.07
1.162	20	14.37	14.09	13.72
1.180	22	15.91	15.61	15.19
1.210	25	18.58	18.23	17.74
1.241	28	21.42	20.99	20.44
1.263	30	23.67	23.21	22.60
1.320	35	28.83	28.28	27.53
Specific Gravity	Degrees Baumé	72% % NaOH	70% % NaOH	60% % NaOH
1.075	10	6.08	5.91	5.06
1.091	12	7.43	7.22	6.19
1.116	15	9.34	9.08	7.78
1.142	18	11.74	11.41	9.78
1.162	20	13.35	12.97	11.12
1.180	22	14.78	14.36	12.31
1.210	25	17.27	16.78	14.38
1.241	28	19.89	19.33	16.57
1.263	30	21.99	21.37	18.32
1.320	35	26.79	26.04	22.31

56. In the preparation of caustic-soda lye in the kettle room, the number of drums of caustic soda and the quantity of water required to furnish a lye of a certain density will be learned by experience. In Table V are given the densities of caustic-soda solutions made from chemically pure caustic soda,

TABLE V

SPECIFIC GRAVITY OF CAUSTIC-SODA SOLUTIONS AT 15° C.
(LUNGE)

Specific Gravity	Baumé	Twaddell	%Na ₂ O	%NaOH	1 cbm. Contains Kilograms	
					Na ₂ O	NaOH
1.007	1	1.4	0.47	0.61	4	6
1.014	2	2.8	0.93	1.20	9	12
1.022	3	4.4	1.55	2.00	16	21
1.029	4	5.8	2.10	2.71	22	28
1.036	5	7.2	2.60	3.35	27	35
1.045	6	9.0	3.10	4.00	32	42
1.052	7	10.4	3.60	4.64	38	49
1.060	8	12.0	4.10	5.29	43	56
1.067	9	13.4	4.55	5.87	49	63
1.075	10	15.0	5.08	6.55	55	70
1.083	11	16.6	5.67	7.31	61	79
1.091	12	18.2	6.20	8.00	68	87
1.100	13	20.0	6.73	8.68	74	95
1.108	14	21.6	7.30	9.42	81	104
1.116	15	23.2	7.80	10.06	87	112
1.125	16	25.0	8.50	10.97	96	123
1.134	17	26.8	9.18	11.84	104	134
1.142	18	28.4	9.80	12.64	112	144
1.152	19	30.4	10.50	13.55	121	156
1.162	20	32.4	11.14	14.37	129	167
1.171	21	34.2	11.73	15.13	137	177
1.180	22	36.0	12.33	15.91	146	188
1.190	23	38.0	13.00	16.77	155	200
1.200	24	40.0	13.70	17.67	164	212
1.210	25	42.0	14.40	18.58	174	225
1.220	26	44.0	15.18	19.58	185	239
1.231	27	46.2	15.96	20.59	196	253
1.241	28	48.2	16.76	21.42	208	266
1.252	29	50.4	17.55	22.64	220	283
1.263	30	52.6	18.35	23.67	232	299
1.274	31	54.8	19.23	24.81	245	316
1.285	32	57.0	20.00	25.80	257	332
1.297	33	59.4	20.80	26.83	270	348
1.308	34	61.6	21.55	27.80	282	364
1.320	35	64.0	22.35	28.83	295	381
1.332	36	66.4	23.20	29.93	309	399
1.345	37	69.0	24.20	31.22	326	420
1.357	38	71.4	25.17	32.47	342	441
1.370	39	74.0	26.12	33.69	359	462
1.383	40	76.6	27.10	34.96	375	483
1.397	41	79.4	28.10	36.25	392	506
1.410	42	82.0	29.05	37.47	410	528
1.424	43	84.8	30.08	38.80	428	553
1.438	44	87.6	31.00	39.99	446	575
1.453	45	90.6	32.10	41.41	466	602
1.468	46	93.6	33.20	42.83	487	629
1.483	47	96.6	34.40	44.38	510	658
1.498	48	99.6	35.70	46.15	535	691
1.514	49	102.8	36.90	47.60	559	721
1.530	50	106.0	38.00	49.02	581	750

57. Caustic Potash.—Potash, or crude potassium carbonate, was originally obtained from the ashes of seaweeds, and before the development of the Le Blanc process it provided the only saponifying agent then available. Later, wood ashes, beet-sugar residues, and wool scourings formed the chief commercial source. The various potassium salts are now obtained chiefly from the mineral carnallite, which is composed of the chlorides of potassium and magnesium with water of crystallization. Carnallite, with other minerals composed of chlorides and sulphates of the alkali and alkaline earth metals, occurs in immense deposits near Stassfurt, in Germany.

Caustic potash is manufactured from carnallite by the Le Blanc process. The potassium chloride is converted into potassium sulphate by treatment with sulphuric acid. A mixture is then obtained which is analogous to the crude Glauber's salt of the Le Blanc process. This is fused with limestone and coal and the resulting mass lixiviated with water. The solution of potassium carbonate thus obtained is causticized with lime, whereby a solution of caustic potash is obtained. This caustic liquor is removed, evaporated to dryness, and fused. While molten it is run into drums and comes into commerce as caustic potash. It is not as uniform in its composition as the corresponding sodium compound. Commercial caustic potash is a mixture of the following substances:

Carbonate and hydrate of potassium....	80 to 95%
Chlorides of sodium and potassium.....	5 to 10%
Sulphate of potassium.....	5 to 15%
Insoluble matter.....	1.5 to 3%

A good caustic potash is generally opaque, of a dull-gray slate or bluish color, often streaked with red or greenish stains. It has a powerful affinity for moisture, and on exposure to the air it deliquesces rapidly and soon becomes pasty. Sometimes it presents a whitish appearance in the center of the drum and is occasionally honeycombed. That which contains a large proportion of salts is usually crystalline

TABLE VI

SPECIFIC GRAVITY OF SOLUTIONS OF CAUSTIC POTASH
AT 15° C. (LUNGE)

Specific Gravity	Baumé	Twaddell	%K ₂ O	%KOH	1 Cbm. Contains Kg.	
					K ₂ O	KOH
1.007	1	1.4	.7	.9	7	9
1.014	2	2.8	1.4	1.7	14	17
1.022	3	4.4	2.2	2.6	22	26
1.029	4	5.8	2.9	3.5	30	36
1.037	5	7.4	3.8	4.5	39	46
1.045	6	9.0	4.7	5.6	49	58
1.052	7	10.4	5.4	6.4	57	67
1.060	8	12.0	6.2	7.4	66	78
1.067	9	13.4	6.9	8.2	74	88
1.075	10	15.0	7.7	9.2	83	99
1.083	11	16.6	8.5	10.1	92	109
1.091	12	18.2	9.2	10.9	100	119
1.100	13	20.0	10.1	12.0	111	133
1.108	14	21.6	10.8	12.9	119	143
1.116	15	23.2	11.6	13.8	129	153
1.125	16	25.0	12.4	14.8	140	167
1.134	17	26.8	13.2	15.7	150	178
1.142	18	28.4	13.9	16.5	159	188
1.152	19	30.4	14.8	17.6	170	203
1.162	20	32.4	15.6	18.6	181	216
1.171	21	34.2	16.4	19.5	192	228
1.180	22	36.0	17.2	20.5	203	242
1.190	23	38.0	18.0	21.4	214	255
1.200	24	40.0	18.8	22.4	226	269
1.210	25	42.0	19.6	23.3	237	282
1.220	26	44.0	20.3	24.2	248	295
1.231	27	46.2	21.1	25.1	260	309
1.241	28	48.2	21.9	26.1	272	324
1.252	29	50.4	22.7	27.0	284	338
1.263	30	52.6	23.5	28.0	297	353
1.274	31	54.8	24.2	28.9	308	368
1.285	32	57.0	25.0	29.8	321	385
1.297	33	59.4	25.8	30.7	335	398
1.308	34	61.6	26.7	31.8	349	416
1.320	35	64.0	27.5	32.7	363	432
1.332	36	66.4	28.3	33.7	377	449
1.345	37	69.0	29.3	34.9	394	469
1.357	38	71.4	30.2	35.9	410	487
1.370	39	74.0	31.0	36.9	425	506
1.383	40	76.6	31.8	37.8	440	523
1.397	41	79.4	32.7	38.9	457	543
1.410	42	82.0	33.5	39.9	472	563
1.424	43	84.8	34.4	40.9	490	582
1.438	44	87.6	35.4	42.1	509	605
1.453	45	90.6	36.5	43.4	530	631
1.468	46	93.6	37.5	44.6	549	655
1.483	47	96.6	38.5	45.8	571	679
1.498	48	99.6	39.6	47.1	593	706
1.514	49	102.8	40.6	48.3	615	731
1.530	50	106.0	41.5	49.4	635	756
1.546	51	109.2	42.5	50.6	655	779
1.563	52	112.6	43.6	51.9	681	811
1.580	53	116.0	44.7	53.2	706	840
1.597	54	119.4	45.8	54.5	731	870
1.615	55	123.0	47.0	55.9	754	902
1.634	56	126.8	48.3	57.5	789	940

and very compact. Its high cost militates against its more general use as a saponifying agent; it is used but little today in soap manufacture. On exposure to the air, its solutions rapidly absorb carbon dioxide with the formation of the acid carbonate. It is a more active chemical agent than caustic soda. Caustic potash improves the quality of all soaps of which it is an ingredient. It cannot be used in settled soap manufacture with sodium chloride as the graining agent. An interchange of alkalis would take place and much of the potassium would be lost as the chloride. Soaps containing it are milder in their deterative action, will stand more filling without efflorescence, and possess a tougher texture.

In virtue of their milder deterative action upon animal and vegetable fibers, potash soaps, usually soft soaps, find wide application in the textile industry.

THE MEASUREMENT OF THE DENSITY OF LIQUIDS

58. Hydrometer.—The specific gravity of a liquid or solid is the expression of the relation between the weight of the liquid or solid and the weight of an equal volume of pure water at a definite temperature, usually 15° C. The most convenient and practical means of ascertaining the strength of solutions of carbonated and caustic alkalis is to determine their specific gravity with an hydrometer. As shown in Fig. 6, the hydrometer is simply a closed tube with a bulb blown in one end, containing a paper scale and filled with shot or mercury. When immersed in a liquid, the weighted bulb enables the hydrometer to float in an upright position. The Baumé and Twaddell hydrometers are the instruments most commonly used, the Baumé hydrometer in America and the Twaddell hydrometer in England. With the latter instrument, the density of pure distilled water is represented by zero and the scale is graduated in such a manner that the specific gravity of the liquid may be calculated by multiplying the

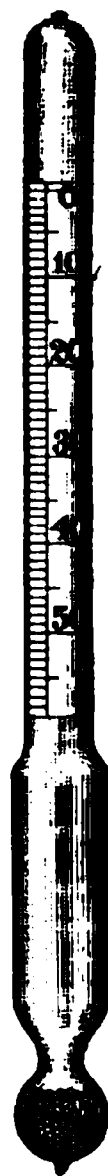


FIG. 6

number of degrees registered on the scale by .005 and adding the product to 1; thus, the density of a liquid indicating 100° Twaddell would be $100 \times .005 + 1.000$, or 1.500. The Twaddell hydrometer is a direct-reading instrument, by

TABLE VII

SPECIFIC GRAVITY AND DEGREES BAUMÉ FOR LIQUIDS
HEAVIER THAN WATER

(Temperature 10° R. = 12.5° C. = 54.5° F.)

Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity
0	1.00000	20	1.15497	39	1.35438	58	1.63701
1	1.00675	21	1.16399	40	1.36680	59	1.65519
2	1.01360	22	1.17316	41	1.37945	60	1.67378
3	1.02054	23	1.18246	42	1.39234	61	1.69279
4	1.02757	24	1.19192	43	1.40547	62	1.71223
5	1.03471	25	1.20153	44	1.41885	63	1.73213
6	1.04194	26	1.21129	45	1.43248	64	1.75250
7	1.04927	27	1.22122	46	1.44638	65	1.77335
8	1.05671	28	1.23131	47	1.46056	66	1.79470
9	1.06426	29	1.24156	48	1.47501	67	1.81657
10	1.07191	30	1.25199	49	1.48975	68	1.83899
11	1.07968	31	1.26260	50	1.50479	69	1.86196
12	1.08755	32	1.27338	51	1.52014	70	1.88551
13	1.09555	33	1.28436	52	1.53580	71	1.90967
14	1.10366	34	1.29552	53	1.55179	72	1.93446
15	1.11189	35	1.30688	54	1.56812	73	1.95989
16	1.12025	36	1.31844	55	1.58479	74	1.98601
17	1.12873	37	1.33021	56	1.60182	75	2.01283
18	1.13735	38	1.34218	57	1.61923	76	2.04038
19	1.14609						

which is meant that the reading on the scale shows the specific gravity of the liquid directly as compared with pure water at the definite temperature.

The Baumé hydrometer is adapted for the determination of the specific gravity of liquids heavier than water and for

liquids lighter than water. The scale used is arbitrary and bears no direct relation to the specific gravity, hence the conversion of the readings into the corresponding specific gravity is done by reference to tables prepared for this purpose.

TABLE VIII

**SPECIFIC GRAVITY AND DEGREES BAUMÉ FOR LIQUIDS
LIGHTER THAN WATER**

Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity	Degrees Baumé	Specific Gravity
10	1.0000	23	.9183	36	.8488	49	.7892
11	.9932	24	.9125	37	.8439	50	.7849
12	.9865	25	.9068	38	.8391	51	.7807
13	.9799	26	.9012	39	.8343	52	.7766
14	.9733	27	.8957	40	.8295	53	.7725
15	.9669	28	.8902	41	.8249	54	.7684
16	.9605	29	.8848	42	.8202	55	.7644
17	.9542	30	.8795	43	.8156	56	.7604
18	.9480	31	.8742	44	.8111	57	.7565
19	.9420	32	.8690	45	.8066	58	.7526
20	.9359	33	.8639	46	.8022	59	.7487
21	.9300	34	.8588	47	.7978	60	.7449
22	.9241	35	.8538	48	.7935	61	.7411

**THE CHEMISTRY OF SOAP
MANUFACTURE**

59. General Remarks.—In the preceding examination of the raw materials of soap manufacture, we have considered those bodies that carry the two compounds, viz., a glyceride and a caustic alkali, which, when in chemical combination, form soap. Soap boiling, therefore, consists essentially in bringing a fatty body and caustic alkali in aqueous solution in contact under suitable conditions,

TABLE IX

SPECIFIC GRAVITY, DEGREES TWADDELL, AND
DEGREES BAUMÉ

Twaddell	Baumé	Specific Gravity	Twaddell	Baumé	Specific Gravity	Twaddell	Baumé	Specific Gravity	Twaddell	Baumé	Specific Gravity
0	0	1.000	44	26.0	1.220	88	44.1	1.440	132	57.4	1.660
1	.7	1.005	45	26.4	1.225	89	44.4	1.445	133	57.7	1.665
2	1.4	1.010	46	26.9	1.230	90	44.8	1.450	134	57.9	1.670
3	2.1	1.015	47	27.4	1.235	91	45.1	1.455	135	58.2	1.675
4	2.7	1.020	48	27.9	1.240	92	45.4	1.460	136	58.4	1.680
5	3.4	1.025	49	28.4	1.245	93	45.8	1.465	137	58.7	1.685
6	4.1	1.030	50	28.8	1.250	94	46.1	1.470	138	58.9	1.690
7	4.7	1.035	51	29.3	1.255	95	46.4	1.475	139	59.2	1.695
8	5.4	1.040	52	29.7	1.260	96	46.8	1.480	140	59.5	1.700
9	6.0	1.045	53	30.2	1.265	97	47.1	1.485	141	59.7	1.705
10	6.7	1.050	54	30.6	1.270	98	47.4	1.490	142	60.0	1.710
11	7.4	1.055	55	31.1	1.275	99	47.8	1.495	143	60.2	1.715
12	8.0	1.060	56	31.5	1.280	100	48.1	1.500	144	60.4	1.720
13	8.7	1.065	57	32.0	1.285	101	48.4	1.505	145	60.6	1.725
14	9.4	1.070	58	32.4	1.290	102	48.7	1.510	146	60.9	1.730
15	10.0	1.075	59	32.8	1.295	103	49.0	1.515	147	61.1	1.735
16	10.6	1.080	60	33.3	1.300	104	49.4	1.520	148	61.4	1.740
17	11.2	1.085	61	33.7	1.305	105	49.7	1.525	149	61.6	1.745
18	11.9	1.090	62	34.2	1.310	106	50.0	1.530	150	61.8	1.750
19	12.4	1.095	63	34.6	1.315	107	50.3	1.535	151	62.1	1.755
20	13.0	1.100	64	35.0	1.320	108	50.6	1.540	152	62.3	1.760
21	13.6	1.105	65	35.4	1.325	109	50.9	1.545	153	62.5	1.765
22	14.2	1.110	66	35.8	1.330	110	51.2	1.550	154	62.8	1.770
23	14.9	1.115	67	36.2	1.335	111	51.5	1.555	155	63.0	1.775
24	15.4	1.120	68	36.6	1.340	112	51.8	1.560	156	63.2	1.780
25	16.0	1.125	69	37.0	1.345	113	52.1	1.565	157	63.5	1.785
26	16.5	1.130	70	37.4	1.350	114	52.4	1.570	158	63.7	1.790
27	17.1	1.135	71	37.8	1.355	115	52.7	1.575	159	64.0	1.795
28	17.7	1.140	72	38.2	1.360	116	53.0	1.580	160	64.2	1.800
29	18.3	1.145	73	38.6	1.365	117	53.3	1.585	161	64.4	1.805
30	18.8	1.150	74	39.0	1.370	118	53.6	1.590	162	64.6	1.810
31	19.3	1.155	75	39.4	1.375	119	53.9	1.595	163	64.8	1.815
32	19.8	1.160	76	39.8	1.380	120	54.1	1.600	164	65.0	1.820
33	20.3	1.165	77	40.1	1.385	121	54.4	1.605	165	65.2	1.825
34	20.9	1.170	78	40.5	1.390	122	54.7	1.610	166	65.5	1.830
35	21.4	1.175	79	40.8	1.395	123	55.0	1.615	167	65.7	1.835
36	22.0	1.180	80	41.2	1.400	124	55.2	1.620	168	65.9	1.840
37	22.5	1.185	81	41.6	1.405	125	55.5	1.625	169	66.1	1.845
38	23.0	1.190	82	42.0	1.410	126	55.8	1.630	170	66.3	1.850
39	23.5	1.195	83	42.3	1.415	127	56.0	1.635	171	66.5	1.855
40	24.0	1.200	84	42.7	1.420	128	56.3	1.640	172	66.7	1.860
41	24.5	1.205	85	43.1	1.425	129	56.6	1.645	173	67.0	1.865
42	25.0	1.210	86	43.4	1.430	130	56.9	1.650			
43	25.5	1.215	87	43.8	1.435	131	57.1	1.655			

TABLE

ACIDS OF THE ACETIC SERIES—GEN

Acid	Formula	Mol. Wt.	Corresponding Glyceride	Formula	Mol. Wt.	Per Cent. of NaOH Absorbed
Formic.....	H	46	Formin		176	66.19
Acetic.....	CH ₃	60	Acetin		318	55.04
Propionic.....	CH ₃ CH ₂	74	Propionin		300	46.15
Butyric.....	CH ₃ CH ₂ CH ₂	88	Butyrin		308	39.74
Valeric.....	CH ₃ CH ₂ CH ₂ CH ₂	102	Valerin		344	34.68
Caproic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂	116	Caproin		356	31.09
Oenanthic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	130	Oenanthin		408	28.04
Caprylic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	144	Caprylin		470	25.54
Pelargonic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	158	Pelargonin		512	23.44
Capric.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	172	Caprin		554	21.06
Undecylic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	186	Undecylin		596	20.14
Lauric.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	200	Laurin		638	18.81
Tridecyllic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	214	Tridecylin		680	17.68
Myristic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	228	Myristin		722	16.53
Pentadecatoic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	242	Pentadecatorin		764	15.70
Palmitic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	256	Palmitin		806	14.98
Daturic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	270	Daturin		848	14.18
Stearic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	284	Stearin		890	13.48
Nondecyllic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	298	Nondecylin		932	12.87
Arachidic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	312	Arachidin		974	12.28
Medullic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	326	Medullin		1,016	11.61
Behenic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	340	Behenin		1,058	11.34
Carnaubic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	354	Carnaubin		1,100	10.61
Hyenic.....	CH ₃ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂ CH ₂	368	Hyenin		1,142	10.14
Cerotic.....	CH ₃ CH ₂	410	Cerotin		1,268	9.46
Melissic.....	CH ₃ CH ₂	452	Melissin		1,304	8.61

ACIDS OF THE ACRYLIC SERIES—GE

Acrylic.....	C ₃ H ₃ ·COOH	72		C ₃ H ₃ (O·C ₃ H ₃ CO) ₂	254	
Crotonic.....	C ₄ H ₅ ·COOH	86		C ₄ H ₅ (O·C ₄ H ₅ CO) ₂	296	
Angelie, Tiglic...	C ₅ H ₇ ·COOH	100		C ₅ H ₇ (O·C ₅ H ₇ CO) ₂	338	25.50
Hypogaric.....	C ₆ H ₉ ·COOH	114		C ₆ H ₉ (O·C ₆ H ₉ CO) ₂	380	15.09
Oleic.....	C ₁₇ H ₃₃ ·COOH	256	Olein	C ₁₇ H ₃₃ (O·C ₁₇ H ₃₃ CO) ₂	884	13.57

ACIDS OF THE LINOLIC SERIES—GI

Elasmargaric....	C ₂₂ H ₃₈ ·COOH	306	Elasmargarin	C ₂₂ H ₃₈ (O·C ₂₂ H ₃₈ CO) ₂	838	14.25
Linolic.....	C ₁₇ H ₃₁ ·COOH	260	Linolein	C ₁₇ H ₃₁ (O·C ₁₇ H ₃₁ CO) ₂	878	13.67

ACIDS OF THE LINOLENIC SERIES—GJ

Linolenic.....	C ₁₇ H ₂₉ ·COOH	278	Linolenin	C ₁₇ H ₂₉ (O·C ₁₇ H ₂₉ CO) ₂	872	13.75
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ACIDS OF THE RECINOLEIC SERIES—GK

Recinoleic.....	C ₁₇ H ₃₁ OH·COOH	298	Recinolein	C ₁₇ H ₃₁ (O·C ₁₇ H ₃₁ OH·CO) ₂	922	13.87
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LE X

GENERAL FORMULA, $C_nH_{2n+1}COOH$

Per Cent. of Glycerine Set Free	Per Cent. of Fatty Acids Set Free	Natural Source
52.28	78.41	The acid occurs in bodies of ants, in the stinging pine, and in certain nettles. Occurs naturally only in slight quantities in oil of <i>Euonymus Europæus</i> .
42.20	82.56	
35.88	86.14	
30.47	87.42	Cow's butter.
26.74	88.94	The acid occurs in the animal and vegetable kingdoms, free, and as an ester. Cow's butter, coconut, and palm-kernel oils.
22.83	90.16	
21.50	91.14	
19.57	91.91	Cow's butter, coconut, and palm-kernel oils.
17.97	92.58	The acid occurs naturally in leaves of <i>pelargonium roseum</i> . Cow's butter, coconut, and palm-kernel oils.
16.61	93.18	
15.44	93.68	Spermaceti, tan oil, coconut, and palm-kernel oils.
14.42	94.06	
13.53	94.41	Spermacetic, muscat butter, coconut, and palm-kernel oils.
12.75	94.75	
12.04	95.11	Associated with stearin and olein in most animal and vegetable fats and oils. Seeds of thorn apple, <i>Datura Stramonium</i> . See palmitin.
11.41	95.20	
10.85	95.52	
10.34	95.73	Characteristic glyceride of peanut oil.
9.87	95.92	
9.44	96.10	Medullic acid is a mixture of palmitic and stearic acids.
9.06	96.27	
8.69	96.40	Characteristic glyceride of oil of <i>BEN</i> .
8.05	96.65	Occurs as an ether in combination with the higher alcohols in carnaūbic wax.
7.77	96.79	Free acid occurs in glandular pouches of the striped hyæna.
7.25	96.99	Acid occurs in free state in beeswax and carnaūbic waxes.
6.60	97.25	Acid occurs in free state in beeswax.

GENERAL FORMULA, $C_nH_{2n-1}COOH$

27.22	88.76	The acid is an oxidation product of acrolein. Acid occurs with iso-crotonic acid in crude pyrolligneous acid. Angelica acid occurs in angelica root. Glyceride occurs in croton oil. Present in peanut oil. The characteristic glyceride of all liquid fats.
11.49	95.26	
10.41	95.70	

GENERAL FORMULA, $C_nH_{2n-1}COOH$

11.01	95.45	Occurs in Japanese wood oil.
10.48	95.68	

GENERAL FORMULA, $C_nH_{2n-1}COOH$

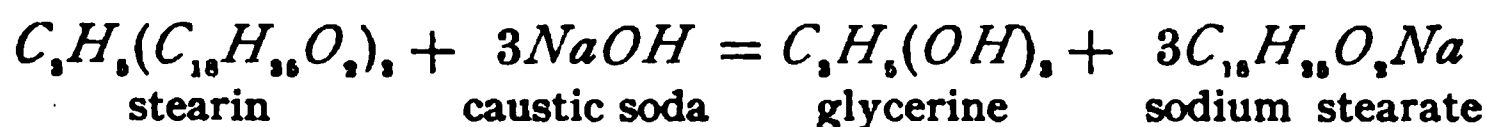
10.55	95.56	Occurs in drying oils, especially linseed oil.
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IS-GENERAL FORMULA, $C_nH_{2n-1}O_2$

9.87	95.92	Characteristic glyceride of castor oil.
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whereby a simple chemical reaction ensues with the formation of an alkaline salt of a fatty acid and the liberation of glycerine. This reaction is known as *saponification*.

60. For the purpose of explanation, the fatty body shall be represented by a molecule of stearin and the saponifying agent by three molecules of caustic soda; thus:



The process of effecting combination between a fat and alkali is called saponification. This term is also applicable to a vast number of similar chemical changes in which an alcohol is set free and an acid is produced, when water is the saponifying agent, or an alkaline salt of the fatty acid, i. e., soap, when the saponifying agent is an alkali.

GLYCERIDES AND THEIR PROPERTIES

61. To thoroughly understand the soap-making properties of the various fats and oils and their behavior in the soap kettle, it is necessary to be familiar with the chemical properties of the various glycerides which constitute the fats and oils employed in soap manufacture. The variation in the amount of alkali absorbed by any particular fat or oil arises from differences in the composition of the glycerides themselves and from the varying proportions in which the glycerides occur in any particular stock. We know that fats and oils are indefinite mixtures of various glycerides, and the amount of alkali absorbed is influenced by the nature of the glycerides characteristic of the stock.

It will be seen from Table X that the amount of alkali required for saturation decreases as the molecular weight of the glycerides increases. Hence, we find that those commercial fats and oils in which the glycerides of low molecular weight occur possess the highest alkali absorption in proportion to their weight.

The greater quantity of salt required for graining the soap made from such stock is due to the presence of those

TABLE XI

Glyceride and Chief Sources	Molec- ular Weight	Yield on Saponifi- cation With Water		Per Cent. Water Absorbed	Yield on Saponifi- cation With Caustic Soda		Per Cent. NaOH Absorbed
		Per Cent. Fatty Acids	Per Cent. Glycerine		Per Cent. Soap	Per Cent. Glycerine	
Laurin—coconut and palm-kernel oils.....	638	94.04	14.42	8.46	104.38	14.42	18.80
Myristin—palm-kernel oils.....	722	94.47	12.70	7.17	103.87	12.70	16.57
Palmitin—palm oil and tallow....	806	95.28	11.41	6.69	103.47	11.41	14.88
Linolein—linseed oil.....	878	95.68	10.48	6.16	103.19	10.48	13.67
Olein—olive and cotton oils.....	884	95.70	10.40	6.10	103.16	10.40	13.56
Stearin—tallow and lard.....	890	95.73	10.34	6.07	103.14	10.34	13.48
Recinolein—castor oil.....	932	95.92	9.88	5.80	103.00	9.88	12.88
Arachidin—peanut oil.....	974	96.10	9.44	5.54	102.87	9.44	12.31

glycerides of low molecular weight whose greater solubility in brine of the sodium salt is a marked characteristic. As the glycerides increase in molecular weight, the solubility in brine of the soap obtained therefrom diminishes; hence, less salt is required for graining.

NOTE.—As shown in *Organic Chemistry*, the homologous character of the simple hydrocarbons of the saturated and unsaturated fatty series extends to all compounds formed from them, either naturally or artificially. The purpose of Table X is to illustrate this property by extending the quantitative characteristics of the more important acids to their corresponding glycerides, and more particularly to those employed in soap manufacture, which appear in heavy type. This table is of great practical interest from the standpoint of both the candle maker and the soap boiler, inasmuch as the most important practical properties of glycerides, viz., alkali absorption and glycerine liberation, are here set forth in detail. It will be seen from the soap-maker's standpoint that to the acetic and acrylic series of fatty bodies belong the most important commercial glycerides, viz., stearin, palmitin, and olein, while to the more unsaturated series belong those glycerides characteristic of all drying oils, which are the special consideration of the paint manufacturer.

62. The homologous properties of the glycerides mentioned in the previous article and shown in Table X, are further shown in their practical application in soap making in Table XI, wherein is indicated the theoretical yield of anhydrous soap from the glycerides mentioned.

63. The reason for the greater yield of soap from coconut oil than from the ordinary animal and vegetable fats is thus made clear. Not only is there a greater yield of anhydrous soap in itself, but in virtue of that higher yield, a greater degree of hydration and "filling" is thus permissible. In Table XII are given the actual and not the theoretical percentages of caustic potash and caustic soda required in practice to saponify the commercial fats and oils mentioned.

BEHAVIOR OF FATS AND OILS TOWARDS SAPONIFYING AGENTS

64. General Remarks.—The various fats and oils that have already been considered display different characteristics in their behavior towards saponifying agents. Moreover, the chemical reactions of organic compounds do not

proceed with the ease and certainty that characterize the reactions of inorganic compounds. In effecting combination between fats or oils and caustic alkalis in a soap kettle,

TABLE XII

Fat or Oil	Per Cent. KOH	Per Cent. $NaOH$
Lard oil.....	19.1-19.6	13.6-14
Almond oil.....	19.5-19.6	13.9-14
Sesame oil.....	19 -19.4	13.5-13.7
Rape oil.....	17.1-17.9	12.2-12.8
Poppy-seed oil.....	19.3-19.5	13.8-14
Niger-seed oil.....	18.9-19.1	13.5-13.6
Whale oil.....	18.8-22.4	13.4-16
Butter.....	22.1-23.2	15.8-16.5
Palm-nut oil.....	22 -24.7	15.7-17.7
Tallow.....	19.3-19.8	13.7-14.1
Sperm oil.....	12.3-14	8.8-10
Palm oil.....	19.6-20.2	14 -14.4
Olive-kernel oil.....	18.8	13.4
Olive oil.....	19.1-19.6	13.6-14
Peanut oil.....	19.1-19.6	13.6-14
Cottonseed oil.....	19.1-19.6	13.6-14
Linseed oil.....	18.7-19.5	13.3-14
Hempseed oil.....	19.3	13.8
Walnut oil.....	19.6	14
Seal oil.....	18.9-19.6	13.5-14
Porpoise oil.....	21.6	15.5
Coconut oil.....	24.6-26.8	17.5-17.7
Lard oil.....	19.2-19.6	13.7-14
Bonefat and grease...	19.1-19.7	13.6-14
Castor oil.....	17.6-18.1	12.5-13
Cod oil.....	18.5-21.3	13.2-15.1
Rosin.....	17 -19.3	12.1-14

where in many instances the fatty bodies are in excess of 100,000 pounds, the chemical peculiarities of the bodies

undergoing change should in every case be thoroughly understood.

As will be explained later, the saponification of a fat is a progressive and step-like reaction, based on its chemical constitution as a triglyceride.

65. In a mixture of fats, the individual fats tend to impart their properties to one another. Certain oils, notably coconut and palm kernel, require caustic lye of high density to effect combination. With others, as tallow and palm oil, saponification ensues readily with caustic lye of lower density. When soap stock with these characteristics are mixed, the requirements of each are modified so that an oil requiring for saponification alone a lye of high density enters into combination with lye of a density with which it would not combine otherwise without time-consuming and troublesome manipulation. On the other hand, that stock requiring naturally for ready saponification a lye of low density enters readily into combination with a lye of higher density. We will consider these peculiarities in the order in which the various fats and oils were originally discussed.

66. Saponification of Tallow.—Tallow requires a caustic lye of comparatively low density for satisfactory saponification. A 12° Baumé lye, when no subsequent dilution is made, is considered to give the best results. On boiling with open steam, a lye of 18° to 20° Baumé is the highest density practicable. When open steam is used with caustic lye of this density, tallow is the most easily saponified of all glyceride soap stocks and the soap formed is most easily grained.

Sodium stearate, which constitutes the bulk of the anhydrous soap made from tallow, is one of the least soluble in water of all the alkaline salts of the fatty acids, it undergoing practically no change when treated with 10 parts of water, neither is its hardness appreciably affected. This quality manifests itself in the inferior lathering properties of pure tallow soap. Furthermore, on aging, a tallow soap becomes

so hard as to be used only with more or less injury to the fabric. The comparative insolubility in water of sodium stearate is also shown in the readiness with which tallow soap dries. Its presence in admixture with other fats imparts firmness or body to the soap, thus enabling a greater proportion of softer stock or that fatty acid whose sodium salt has a greater affinity for water, to be incorporated. Its use thus increases the amount of water that may be incorporated with a soap without an excessive sacrifice of firmness. It is the best raw material for the manufacture of grained soaps and admits of the greatest yield of all the animal soap stocks. With its use, the various chemical and physical changes occurring in the soap kettle during the process of boiling are well defined and of easy recognition.

67. In Table XIII is expressed the saponifying power of caustic lyes of different densities, made from chemically pure and from the ordinary grades of caustic occurring in the market. By reference to this table, the amount of stock that can be saponified by any quantity of lye of the density and quality given may be readily determined within limits of accuracy suitable for all technical purposes to which the table may be applicable.

68. Saponification of Cottonseed Oil.—Refined cottonseed oil saponifies with difficulty and only after continued boiling, especially when saponified alone. The absence of free fatty acids tends to retard saponification. Combination with alkali is hastened when tallow is present, or on the addition of soap scraps. Saponification is best begun with a 15° Baumé lye.

Pure cottonseed oil soap is white, with a firmness determined largely by its degree of hydration. It consists almost entirely of sodium oleate. This compound is soluble in 10 parts of water, while sodium stearate, as we have seen, is not appreciably affected by this volume. In accordance with its greater solubility in water, we note, in comparison, its greater and peculiar lathering properties. It lathers

more readily than does sodium stearate, but instead of the firm lather of tallow soap, obtained only after much rubbing, we have a shiny, thin lather peculiar to all soaps made of stock consisting largely of olein, viz., Castile soap and cottonseed-oil soap stock. Cottonseed oil is generally used in admixture with varying proportions of tallow and grease.

TABLE XIII

POUNDS OF TALLOW SAPONIFIED BY 100 LB. OF CAUSTIC SODA OF DIFFERENT DENSITIES MADE FROM VARIOUS GRADES OF CAUSTIC

Density, Degrees Baumé	Grade of Caustic Soda					
	77½% Na_2O	76% Na_2O	74% Na_2O	72% Na_2O	70% Na_2O	60% Na_2O
	Lb. Tallow Saponified	Lb. Tallow Saponified	Lb. Tallow Saponified	Lb. Tallow Saponified	Lb. Tallow Saponified	Lb. Tallow Saponified
10	46.78	45.86	44.66	43.45	42.24	36.20
12	57.14	56.03	54.55	53.08	51.59	48.49
15	71.85	70.46	68.60	66.74	64.88	60.96
18	90.02	88.26	85.94	83.62	81.28	69.66
20	102.64	100.60	97.95	95.30	92.64	79.39
22	113.64	111.40	108.50	105.50	102.60	87.92
25	132.71	130.10	126.70	123.30	119.90	102.70
28	153.00	150.10	146.10	142.10	138.10	118.40
30	169.07	165.80	161.40	157.00	152.60	130.80
35	205.92	201.90	196.60	191.30	186.00	159.30

A good settled soap of cottonseed oil alone is not practicable. Soap thus made is "thin" and lacks the body and durability of a tallow soap or a soap made from mixed stock. By the use, however, of a large proportion of soda ash and sodium silicate, an artificial firmness may be imparted.

Cottonseed-oil soaps sweat readily, and unless the oil has been refined with care, discolors rapidly and becomes rancid. The brownish or yellowish blotches seen in soaps containing

cottonseed oil may be traced to the imperfect removal of the coloring matter of the seed during the refining process.

69. Saponification of Coconut and Palm-Kernel Oils.—Coconut and palm-kernel oils differ from all fatty bodies employed in soap manufacture in the greater quantity of alkali required for saponification, in the greater quantity of salt required for graining, and in the greater yield of soap produced and of a quality that permits the greatest amount of hydration and filling.

Pure coconut-oil soap is white, brittle, and hard. The oil is a valuable addition to cottonseed oil and tallow and yields a soap possessing qualities superior to that possessed by either stock alone. It enters into combination with alkali with extreme ease, which property especially adapts it for soap manufacture by the cold process.

During saponification considerable heat is evolved, and saponification when once begun ensues with such rapidity that the contents of the kettle, unless carefully watched, will boil over. On the stock change (stock saponification), difficulty is often experienced in working with coconut oil, or a mixture of oils containing coconut, in the soap mass becoming thick. This is due to the high absorption of water consequent upon rapid saponification and is overcome by retarding the rate of combination of caustic alkali and oil. This is best effected by the addition of brine when the soap mass shows signs of becoming thick. It may also be avoided by adding the oil slowly to the caustic alkali already in the kettle, at the same time boiling vigorously.

70. Coconut oil combines with weak and strong caustic lyes with equal facility, but in practice in the boiling of settled soap, a density of from 20° to 25° Baumé is commonly used. Coconut-oil soap when hot is very fluid, smooth, and transparent; when cold, it becomes so hard as to be cut only with difficulty. It dissolves readily in water, yielding a quickly formed, profuse, but not permanent lather. It is caustic to the tongue even though no free alkali is present, and when used for toilet purposes in excess of actual

requirements excoriates and reddens the skin. If coconut oil is not completely saponified, the soap made from it soon becomes rancid and odorous. It dissolves freely in salt water and is the fatty basis of the so-called marine, or salt-water, soaps.

71. The complex composition of coconut oil suffices to explain the peculiarities of its behavior towards saponifying agents and the properties of the soap made from it. Coconut and palm-kernel oils contain a greater variety and quantity of glycerides of fatty acids of low molecular weight than any other soap stock.

As will be seen by reference to Table XI, these bodies possess a higher alkali absorption, yield a greater percentage of glycerine on saponification, are more soluble in water when free and combined with alkali, and in general possess less stable chemical properties than do similar bodies of higher molecular weight which occur commercially as soap stock.

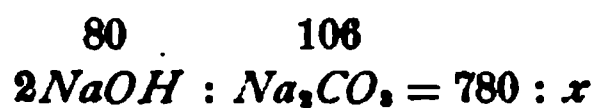
72. Saponification of Palm Oil.—Palm oil, of which palmitin is the chief glyceride, is of limited use as a soap stock in this country. It is used chiefly as an ingredient of certain toilet-soap bases (for which purpose it is well adapted by its perfume) and to disguise the odor of rosin in excessively rosined soap. In Great Britain and Continental Europe it replaces tallow to a large degree.

Sodium palmitate so closely resembles sodium stearate in its chemical properties that the statements concerning the properties of the latter salt are directly applicable to it. Palm oil saponifies very readily, owing to the high percentage of free fatty acids characteristic of the oil.

73. Saponification of Rosin or Colophony.—Rosin when in a pure state consists of the anhydrides of the rosinic acids, chiefly abietic acid $C_{10}H_{17}(CO_2H)$. On boiling it is readily transformed into the acid which combines with both carbonated and free alkali. In ordinary practice it is

seldom "killed" or saponified alone, but added after the kettle has been charged with tallow, the latter either saponified previously or both stocks killed together. Also, in ordinary practice, soda ash is seldom used as the saponifying agent. The rosin soap thus made is frothy from the liberation of carbonic-acid gas, and as a result of the increased volume caused thereby, much kettle space is required to work it. However, where factory conditions are such as to warrant the use of soda ash instead of caustic soda, a considerable saving is possible, as is shown in the following approximate calculation:

EXAMPLE.— 6,000 pounds rosin $\times .13 = 780$ pounds (approx.) of 74-per-cent. (see table XIII) caustic soda required for combination. $\frac{74}{100} \times \$1.50 = \1.85 , cost per 100 pounds of 74-per-cent. caustic soda when price is \$1.50 per 100 pounds for 60 per cent. $\$1.85 \times 7.8$ hundredweight = \$14.43, cost of 74-per-cent. caustic soda, required for combination.



$x = 1,033.5$ pounds soda ash required for combination.

$1,033.5 \times \$0.007 = \7.23 , cost of soda ash required for combination.

Caustic soda costs	\$14.43
Soda ash costs	7.23

Saving in alkali for 6,000 pounds rosin.....\$7.20

74. The alkali salts of the rosin acids are very hygroscopic and naturally readily soluble in water and, as we should expect, make a very soft soap. They detract from the firmness of all soaps in which they enter. Rosin betrays its presence in the finished soap by the darker color and by its characteristic odor and stickiness, which latter property is due to the marked affinity of the alkaline resinsates for moisture. This peculiarity manifests itself in the readiness with which all rosined soaps sweat. The marked detergent property, ready solubility in either hot or cold, soft or permanently hard water, and cheapness of the alkaline resinsates, make them an indispensable addition to the firmer tallow soap for domestic purposes. The characteristic odor may be

ameliorated by the addition of suitable perfumes, and the stickiness reduced by the admixture in reasonable proportions of the tallow and rosin base. All rosined soaps become very dark on aging.

75. The best proportions of tallow and rosin to employ depend primarily on the firmness of the tallow, i. e., on the percentage of stearin present. A soft tallow cannot assimilate as much rosin as a firm tallow and the desired firmness of the finished soap be obtained.

It has been the custom of the trade to rate rosined soaps according to the proportion which the rosin bears to the fat stock, by what is known as soap-makers' percentage. A 100-per-cent. rosin soap contains equal parts of tallow and rosin; a 50-per-cent. rosin soap contains 3 parts tallow and 1 part rosin; a 150-per-cent. rosin soap contains 1 part tallow and 3 parts rosin.

A more rational method of expressing the proportions is the simple percentage of rosin on the basis of 100 pounds of fat stock used. Thus, a mixture of 3 parts fat and 1 part rosin, which by soap-makers' percentage is called a 50-per-cent. rosin soap, is equivalent to 100 pounds of fat stock and $33\frac{1}{3}$ pounds of rosin, or as simply and more easily understood, a 33 $\frac{1}{3}$ -per-cent. rosin soap.

A soap properly made of these ingredients and in the proportions just stated, with not more than 8 per cent. of soda-ash solution added in the crutcher, constitutes the standard high-grade settled rosin soap.

76. Saponification of Olive, Red, Corn, and Cottonseed Oils.—These oils possess the same general chemical characteristics and behave in the same manner towards saponifying agents.

Olive oil was originally the basis of the soap known to Americans as *Castile soap* and on the Continent of Europe as *Marseilles soap*. Pure olive-oil soap is white and very mild in its deterative action. It is composed chiefly, if caustic soda is used to effect saponification, of sodium oleate. It is

an excellent ingredient of a toilet-soap base and is especially adapted to use in the textile industry.

Red oil, or commercial oleic acid, being a stronger acid than carbon dioxide, can be saponified with soda ash. Red-oil soap, sometimes called *oleine soap*, is practically identical in its composition and properties with olive-oil soap and is therefore equally adapted to the cleansing of textiles.

Cottonseed-oil soap stock, when in a clean condition and as free as possible from coloring matter, is identical in composition with red-oil soap and is adapted to the same technical uses. It must be remembered, in connection with the use of cottonseed-oil soap stock, that it is simply soap, and unless in a very dehydrated condition does not admit of the yield common to all glyceride stock. It is simply an addition used because of its cheapness.

77. Removal of Stock From Barrels.—The various animal and vegetable soap stocks are most quickly and conveniently removed from their containers by blowing in live steam. The casks are rolled upon a track over the melting trough, shown in Fig. 7, and a swing-joint steam pipe inserted in the bung hole. The melting trough is made of



FIG. 7

sheet steel, and when the sides serve to support the casks, they are strengthened with a band around the top. The live steam quickly melts the stock, which flows into a storage tank. Here it is left to settle out any foreign matter, or it may be pumped or blown directly to the soap kettle or given a preliminary bleaching.

PROCESSES OF SOAP MANUFACTURE

78. General Remarks.—As we have seen, soap boiling consists essentially of effecting a chemical reaction between a fatty body and a caustic alkali. The manner of effecting this combination and the conditions under which the reaction is completed give rise to three general classes of soap-manufacturing processes, viz., boiled, semi-boiled, and cold processes, whereby soap, to which the same descriptive terms may be applied as well, are produced.

Boiled soaps are also called *settled soaps* when in process of manufacture they have been subjected to changes whereby the soap is purified and the glycerine separated.

A *semi-boiled* soap is one that has not been subjected to a graining process, but contains all the material added to the kettle. It is also called a *run soap*, which term, however, is without technical significance.

A *cold soap* is one made by the direct combination of the materials in the proportions in which they are to remain in the finished soap, the combination being effected without the aid of heat other than that required to bring the ingredients to the requisite temperature and that heat evolved by the chemical reaction.

The classification of processes just given is arbitrary and is not based on any essential chemical differences in the processes. The division is more mechanical than chemical and has reference chiefly to the time required, the artificial heat employed, and the mechanical apparatus necessary to a satisfactory operation of the process.

THE MANUFACTURE OF BOILED OR SETTLED SOAPS

79. Boiled Soaps.—These soaps are the most important and constitute the class most generally manufactured and used. All household soaps are made by this process as well as the base for toilet soaps.

Before discussing in detail the manufacture of soap of this class, we will consider the essential features of the process as it is continued throughout the time required for its completion. There is arranged in Table XIV what we may call "The Chronology of a Boil of Settled Rosin Soap." In this table is embodied an outline of the most important details of a boil of soap of this character. It is the soap most commonly made in this country, and the procedure here outlined is the one generally employed.

In an elaboration of the treatment given the soap in the kettle, we shall discuss thoroughly the directions and precautions to be observed from the addition of the soap stock and alkali to the kettle, through the various changes to the transference of the product to the crutcher and the addition of filling agents, to its final treatment in the drying room and its preparation for shipment.

TABLE XIV

CHRONOLOGY OF A BOIL OF SETTLED ROSIN SOAP

Chemical or Physical Change	Description of Process		Time
	Material Added to Kettle	Material Added to and Withdrawn from Kettle	
Saponification or Stock Change	Straight stock or a mixture of tallow, grease, and cottonseed oil, previously steamed from packages, is pumped from storage tanks to the soap kettle.	Solid caustic soda of 74° quality is dissolved in water and reduced to 20° Baumé. This lye is kept in storage tanks from which it is pumped as required.	First Day
	Stock and lye are run into kettle together and maintained in vigorous ebullition with open steam, before and during saponification, until the greater part of the stock has been added. The completion of saponification and obtaining soap without caustic taste require great care and experience.		
Graining	Either fresh salt, saturated brine (25° Baumé), or dry salt recovered from waste lye in glycerine refinery is added in quantity sufficient to bring soap to the proper "grain." Soap made from a suitable admixture of tallow, grease, and cottonseed oil may be considered grained when waste lye withdrawn contains 7-10% salt.		

TABLE IV—(Continued)

Rdsin Change		After settling overnight the under lye is withdrawn. This is the stock lye and is richest in glycerine. It is pumped to the glycerine refinery.	Second Day
	Pulverized rosin is added to kettle with addition of caustic lye as required. Boiling is continued throughout the day. The soap is kept "open" on the rosin change and grained "close" or "hard." A hard grain at this stage greatly facilitates the dropping of the impurities present in the rosin.	In order to more completely remove the glycerine from the killed stock, the rosin change may be preceded by a "pickle" change, whereby the soap is boiled on brine sufficient to keep it grained and settled again over night.	
Strengthening Change		After settling over night the under lye is withdrawn. This is the rosin lye and is pumped to the glycerine refinery.	Third Day
	Contents of kettle are boiled up and alkali added in amount sufficient to keep soap open. Boiling is continued throughout greater part of the day. This is the most important change of the entire boil. As the alkali is absorbed, more must be added to retain soap in grained condition, in which state it is allowed to settle over night. The alkaline treatment received on this change is most effective in removing impurities and the salt retained mechanically from preceding changes.		
Fitting the Soap	After running off the strength lye, steam is turned on. When soap comes to a boil, water is added and the soap brought to a proper "finish."		Fourth Day
Settling the Niger	The soap is now left to "drop" the niger and to cool. A maximum period of 5 days is required to settle out the impurities, although this period varies with the size of the kettle and the season. The strength lye containing considerable alkali is reserved in storage and utilized on the "niger" change of a new boiling.		Fifth, Sixth, Seventh, Eighth, and Ninth Days
Crutch- ing Soap	Hot, liquid soap is pumped from kettle to the crutcher, where it is mixed with soda ash, sodium silicate, borax solution, perfumery, etc. Soap is then dropped into frames and allowed to cool and solidify.		Tenth Day

TABLE XIV—(Continued)

Niger Change	The strength lye is added to the alkaline niger and grease run in. Boiling is continued vigorously until all strength is exhausted. As the soap at this stage is very dark colored from impurities present in the niger and strengthening lye, it is best to grain sharply to remove them before adding fresh stock. The utilization of the niger method varies greatly and is determined largely by circumstance. It is often transferred to another kettle containing soap on the rosin change, or, as is more commonly the practice, a new boil is started on it.	Three days usually suffice for soap in frames to arrive at condition suitable for slabbing and cutting, after which the soap is dried, pressed, wrapped, and boxed.	Eleventh Day
Stock Change (New Boil)	Niger lye is withdrawn and pumped to glycerine refinery. To the grained niger fresh stock is added and a new boil begun.		Twelfth Day

80. The Soap Kettle.—The soap kettle is made of sheet steel of a thickness determined by the capacity and carefully riveted. The kettle is well supported both at the bottom and at the sides. The shape generally used today is either cylindrical, or slightly truncated as shown in Fig. 8, with cone-shaped or dished bottom, or square or rectangular in cross-section, with the bottom either sloping to one side, or from all sides towards the center. The circular or square cross-section is largely a matter of personal preference, both shapes being equally efficient when properly provided with steam coils. Kettles of square cross-section are more economical of space and allow the architect to design a kettle room that is more easily kept clean. It is necessary to brace square kettles with cross-rods; with the cylindrical kettle this support is not required. The kettle is provided with a swing-joint pipe 3 inches in diameter through which the liquid soap is removed by pumping. The location of the swing-joint pipe is determined by the dimensions of the kettle and should be at that height which will permit the end of the pipe to touch the lowest point in the bottom of the kettle and with the other, or pivot end, above the niger line. The swing-joint pipe is shown at *d* in Figs. 8 and 9.

81. Waste lye is removed at *c*, as shown in Figs. 8 and 9, at the bottom in the case of the cylindrical kettle and from the lowest point on the side near the bottom in the case of the square kettle, the bottom of which slopes to the side.

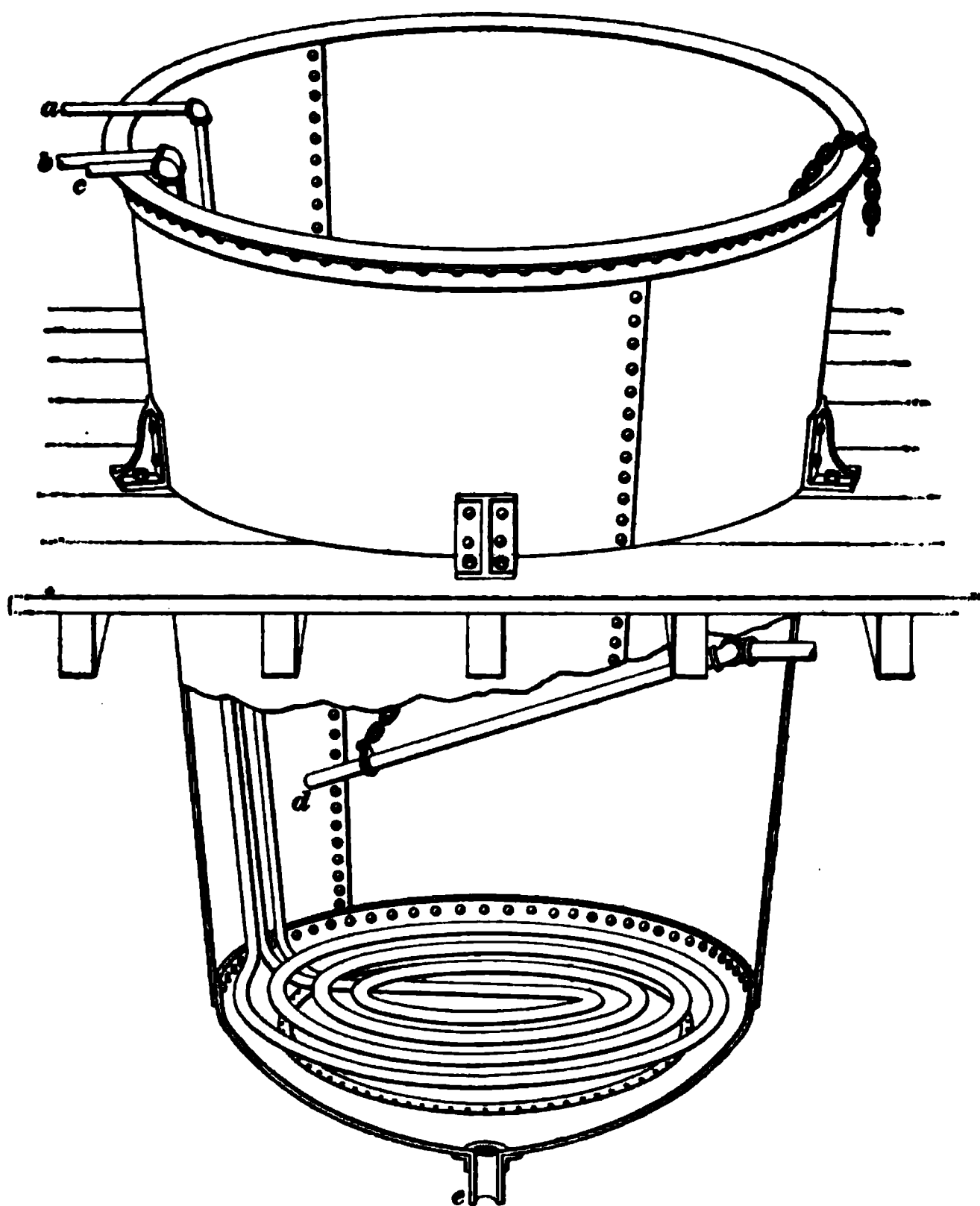


FIG. 8

The waste-lye pipe should not be less than 3 inches in diameter, and the opening to it should be covered with a perforated iron plate to serve as a strainer.

82. The steam piping in a soap kettle is a very important matter, for the successful handling of the contents of the kettle very largely depends on the shape, diameter, and

distribution of the coil. The diameter of the kettle will determine whether one or two open steam coils shall be used. * With a small kettle, one open steam coil or crisscross jet in the center will suffice. This crisscross jet is shown at *f* in Fig. 9. With a kettle 15 to 20 feet in diameter, a second open steam coil around the sides is necessary. The closed steam coil should completely cover the bottom of the kettle. Such

FIG. 9

a coil, with an open steam coil in the center, is shown in Fig. 10, in which *a* represents the central open steam coil, and *b* and *c*, respectively, the inlet and outlet of the closed steam coil. In Fig. 8, *a* represents the open steam coil located at the middle of the sides of the dished bottom, and *b* and *c*, respectively, the inlet and outlet of the closed steam coil. The single-pipe coil for closed steam, shown in Figs. 8

and 10, is more expensive than the jointed coil shown in Fig. 9, but is without leakage. The jointed steam coil is cheaper, more quickly and easily installed, and may be enlarged or repaired with but little trouble and expense. The only disadvantage is the greater possibility of leakage. The valves on both the open and closed steam coils should be within easy reach of the soap boiler.

83. The amount of stock that can be killed in a kettle of given capacity varies with the method of working. In general terms it may be stated

FIG. 10

that 1 cubic foot is allowed for every 15 pounds of stock to be saponified, or for every 100 pounds of stock, a capacity of $37\frac{1}{2}$ gallons is required. * In Table XV are given the dimensions of soap kettles corresponding to the capacities given.

84. Saponification, or the Stock Change.—In this discussion we shall use the following combination of stock that will produce a laundry soap of good standard quality and that will represent the typical stocks employed in American practice in the boiling of settled rosin soap.

Stock Ingredients	Pounds	Percentage
Tallow.....	11,250	50
Bone stock or grease.....	7,500	$33\frac{1}{2}$
Cottonseed oil.....	3,750	$16\frac{1}{2}$

TABLE XV

DIMENSIONS AND CAPACITIES OF SOAP KETTLES

Capacity in gallons.	150	300	450	600	750	900	1,050	1,200	1,500	1,800	1,875	2,250	2,700	3,000	3,750	4,500	5,000	6,000	7,500	9,000	10,000
Diameter at top in inches.....	42	54	60	66	72	78	84	90	96	96	96	96	108	108	108	108	120	126	138	144	150
Diameter at bottom in inches.....	30	42	48	54	60	66	72	78	84	84	84	84	84	84	84	84	96	114	114	120	126
Depth in inches.....	36	42	48	54	60	66	72	78	84	84	84	84	84	84	108	126	120	126	132	144	150

These proportions may be modified according to the prevailing market price for the different ingredients and the quality of soap desired.

85. By reference to Table XII, it is seen that the alkali absorption of these three stocks is practically identical, viz., 14 per cent. of chemically pure caustic soda. We will accept the figures given in Table IV, where we learn that a 20° Baumé caustic-soda lye, made from 74-per-cent. caustic, contains 13.72 per cent. of sodium hydrate. Caustic-soda lye of 20° Baumé is the density commonly employed, and 74-per-cent. commercial caustic, we have learned, is about the cheapest grade to buy. We see at once that for all practical purposes, 1 pound of 20° Baumé caustic-soda lye will kill 1 pound of stock. We are able to confirm this by referring to Table XIII, where we find that 100 pounds of caustic-soda lye of 20° Baumé, made from 74-per-cent. caustic, will kill 97.95 pounds of tallow.

With the variation prevailing in the quality of the raw materials, both organic and

inorganic, used in the manufacture of soap, we cannot calculate with accuracy. We can ascertain the quantity of alkali theoretically absorbed by a given stock and use the quantity so calculated, but we will not infrequently find that our soap will contain either a greater or less quantity of free alkali or more or less unsaponified matter, and unless we have practical experience in soap boiling, it is almost certain that our soap will contain both.

Experience gained by work over the soap kettle is the final arbiter. The conscientious man will use the calculations of theory and supplement them with the tests of his senses. It is best to have a small quantity of unkilld stock on the stock change and the alkali completely absorbed than an excess of alkali wasted in the stock lye.

86. Stages of Saponification.—The saponification of glyceride stock requires three stages for the complete formation of glycerine and combination of the alkali with the fatty acids. While these stages do not admit of exact definition in the soap kettle, they manifest themselves in certain characteristic conditions, which are familiar to every soap boiler, viz., the emulsion formed on admixture of stock and lye, the pasty mass obtained on continued boiling, and, lastly, the final condition resulting from boiling the pasty mass with an amount of alkali sufficient for complete saponification. By means of chemical formulas, we are able to graphically represent the three successive stages of saponification:

Raw Materials:

Stearin (tallow) $C_2H_5(C_{18}H_{35}O_2)_2$	Caustic soda: $3NaOH$
I. Emulsion $C_2H_5OH(C_{18}H_{35}O_2)_2$	Soap: $C_{18}H_{35}O \cdot NaO$
	Caustic soda: $2NaOH$
II. Pasty mass $C_2H_5(OH)_2(C_{18}H_{35}O_2)$	Soap: $2C_{18}H_{35}O \cdot NaO$
	Caustic soda: $NaOH$
III. Glycerine $C_3H_5(OH)_3$	Soap: $3C_{18}H_{35}O \cdot NaO$

87. If the piping of the kettle room permit, all three stocks indicated in Art. 84 may be run in together;

however, this is of minor importance, the chief care being to add the stock and alkali simultaneously with vigorous boiling, in such proportions that after saponification has started, the mass is kept well open until the greater part of the alkali has been added.

While the alkali and stock are being run in together, the mass in the kettle must be kept homogeneous by vigorous agitation or boiling with live steam. Unless maintained in agitation, too little alkali is apt to cause "bunching," by which is meant local saponification enclosing a mass of unkilld stock; vigorous boiling disintegrates and distributes this mass.

An excess of alkali grains the mass, thus throwing it, as it were, out of solution, whereby combination is retarded.

By the time the total amount of stock has been added, the greater part of the alkali should also be in the kettle and boiling is continued, though less vigorously than at the beginning.

88. Care should be exercised to avoid an excess of alkali, and at the end of the change it should be added in small quantities only and not until the strength of the quantity added previously has been absorbed. When the stock has been completely killed, the soap should slide freely from the paddle in large, transparent flakes. A small portion rubbed between the fingers should curl up smooth and dry and without any indication of grease. At this stage the contents of the kettle should boil smoothly, rising in the middle and descending at the sides, the appearance being very characteristic.

On the completion of saponification, the contents of the kettle has assumed a clear, homogeneous mass, in which is present everything that has been added during this stage, viz., soap, glycerine, water, and some sodium chloride, sodium sulphate, and sodium carbonate, introduced as impurities with the caustic-soda lye. In addition, there is more or less mucilaginous or albuminous matter that was present as animal tissue in the stock.

89. Graining the Soap.—The separation of soap from water is based on its insolubility in solutions of salts and

alkalis. In practice, common salt, either in a dry state or as brine, is used for this purpose. • If an excessive quantity of water is present in the kettle, either from condensation of the steam used in boiling or from the use of weak caustic-soda lye, dry salt should be used.

Where open steam is used for boiling, more concentrated solutions, both of caustic soda and common salt, must be used than would be required with the use of closed steam, consequently, the working capacity of the kettle is not only considerably reduced, but the contents is made unsatisfactory to work. Caustic-soda lye of a density of 20° Baumé is entirely too concentrated to saponify tallow. In fact, with caustic lye of such a strength, without dilution, saponification of animal and most vegetable soap stock is impracticable. It is more convenient and more satisfactory to prepare concentrated caustic lye and to dilute it with water as it is run into the kettle.

90. An excessive quantity of water in the kettle manifests itself in frothing, and under such conditions dry salt must be used. • The nature of the stock used should also be considered. Tallow alone forms a soap which at this stage is of a characteristically firm consistency, although fluid.

Soaps made from cottonseed oil, red oil, olive oil, and rosin are very fluid, and from their consistency would seem to indicate the presence of an excessive quantity of water when such is not the case. Coconut oil makes a very fluid soap, and because of the large amount of salt required for graining, the latter should invariably be added in the dry state. •

91. The purpose of graining is to separate the soap from the superfluous water, with which it is associated and which, forming the menstruum for the salt, glycerine, impurities in the stock, lye, etc., constitutes the stock lye. •

With the contents of the kettle boiling quietly, dry salt or saturated brine is added in small quantities at a time and thoroughly boiled through the soap until a portion taken up on a paddle coagulates or separates so that waste lye runs from it. The waste should be clear, of salty taste, and

should not contain free alkali in excess of four-tenths of 1 per cent. This amount of free alkali in waste-soap lye is not perceptible to the taste.

The desired consistency of the grain is a condition arrived at by experience. The use of salt in excess of the amount required to produce this is not only wasteful, but also, if not thoroughly removed in the final settling change, tends to make the soap become fissured or "cracky" in the frames. The amount of salt added will determine the degree of hydration of the grained soap.

The more concentrated the salt solution in contact with the soap, the less water will be retained by the soap. With an insufficient quantity of salt, the soap will not be withdrawn entirely from solution. With such a condition, the contents of the kettle on cooling will not present a clean line of demarcation between the soap and the under lye, but there will be an intermediate zone of soft soap. Waste lye withdrawn hot from soap that has been insufficiently grained, will, on cooling, have the consistency of soft soap.

92. In the boiling of soap of the character that we are discussing, the stock lye should not have a greater density than 13° Baumé, and will contain from 7 to 10 per cent. of salt. The soap is sufficiently grained when a waste lye of this character is withdrawn. It has been found by analysis that this is the lowest density that will remove the soap completely from solution; more salt than this quantity is unnecessary and wasteful. The stock lye is the clearest and least discolored and is the most valuable, owing to the high percentage of glycerine present.

93. With the first appearance of a tendency to grain, the quantity of salt added previously should be thoroughly boiled through the soap and each subsequent addition should be boiled through in the same manner before more is added. When the desired grain has been obtained, the soap is boiled up to the top of the kettle, steam is turned off, and the contents allowed to rest until the following morning.

* During this time, the soap, in virtue of its lower specific gravity, will have risen to the top and separated completely from the brine.

When the waste lye is withdrawn on the following morning, the soap will subside in the kettle an amount equal to the volume occupied by the waste lye. The appearance, shown on the sides of the kettle, should be noted, in the effort to obtain uniformity of treatment of successive boils.

94. Rosin Change.—In the boiling of settled rosin soap, this change is the one most easily made. After the stock lye has been withdrawn, open and closed steam are turned on, and the greater part of the caustic alkali, required to kill the weight of rosin used, is run in. • The heads and staves of the rosin barrels should have been previously removed and the rosin broken up upon bars, between which it falls into a cart or barrow below; it is added to the kettle through a trap door in the floor immediately above. Where this convenient method is not practicable, the rosin can be broken up on the kettle-room floor and shoveled directly into the kettle. This method, however, entails considerable work in keeping the kettle room clean. Caustic-soda lye of 20° Baumé is also used on this change. The rosin should be broken into pieces not larger than the double fist. Unless broken into small pieces, it sinks immediately to the bottom of the kettle, where, if the kettle has a cone-shaped bottom and is not adequately provided with steam coils, the rosin melts very slowly and the procedure is unnecessarily prolonged.

On the addition of alkali, as previously noted, the soap is well boiled through, simultaneously with the addition of the rosin. Combination ensues quickly. The same care must be taken to insure the fullest absorption of the alkali, with the least amount added in excess to be discharged into the rosin lye.

95. During this change combination is facilitated by keeping the contents of the kettle in a state of partial precipitation by the addition of caustic-soda lye. • In the language

of the soap boiler, this is termed "graining the soap with alkali," so called from the consistency which the soap assumes, or "keeping the soap open on alkali," by which is meant not allowing the soap to "close" or to lose its grainy appearance on passing into complete solution.

"Opening the soap," therefore, means partially precipitating it by the addition of a body in whose solution it is insoluble.

Towards the end of the change, while the last traces of strength are being absorbed, to avoid the possibility of an excess of alkali, dry salt can be used for this purpose. This not only allows the contents of the kettle to be worked more easily, but enables a more complete discharge of the coloring matter present in the rosin.

After all the alkali that can be absorbed is added and no traces of it are perceptible in the waste lye, the graining of the soap mass is completed in precisely the same manner as outlined in Art. 89. The same remarks relative to the stock lye are also directly applicable to the rosin lye.

After the requisite amount of salt has been added to bring the soap mass to the desired grain, the boiling is continued until it rises up to the top of the kettle. The contents of the kettle is now allowed to rest until the following morning, during which time the rosin lye separates.

96. The rosin lye is invariably very highly colored, with a depth of color depending largely on the quality of the rosin used.

On the stock change, 22,500 pounds of mixed stock were killed. This admixture will carry well one-third its weight, or 7,500 pounds, of rosin—roughly 15 barrels. After the withdrawal of the rosin lye we will have in the kettle about 45,000 pounds of soap.

97. Strengthening Change.—After the rosin lye has been withdrawn, open and closed steam is turned on. Water is added in just sufficient quantity to close the soap, that is, to transform the contents of the kettle into a homogeneous mass without the separation of soap and lye (see

Art. 95). With the soap boiling smoothly, 20° Baumé alkali is added gradually with sufficient water to dilute it to 8° or 10° Baumé until the soap is just barely open. (For explanation of this term see Art. 95.) With the soap maintained in this condition, by the addition of successively small quantities of caustic lye, as it is absorbed and the soap tends to close, boiling is continued throughout the greater part of the day. After each addition of alkali, the soap will tend to close both by the absorption of the alkali and by the dilution of the water from the condensed steam. More alkali is then added until the soap is slightly open as before and the procedure repeated until the attendant is satisfied that the absorption of alkali by the stock is complete. Towards the end of the change the soap is boiled up to the top of the kettle. At this stage, the soap should be just open and have a good sharpness or alkalinity, although by no means excessive. The soap is now in a good condition for settling. Steam is turned off and the contents of the kettle is allowed to stand until the following morning.

98. As noted on the stock and rosin changes, to avoid an excessive quantity of free alkali being discharged into the waste lye withdrawn from these changes, it is much better to leave the stock incompletely killed and no free alkali in the waste lye, than to have the stock thoroughly killed with an excess of alkali present.

The purpose of the strengthening change is to complete saponification and to discharge the salt, coloring matter, and other impurities retained mechanically by the soap. No salt is used for graining on this change. The 3 to 6 per cent. of salt commonly present in strengthening lyes represents the amount that has been retained mechanically by the soap and washed from it on this change.

The alkali wash promotes the discharge of the coloring matter, and completing the saponification of the last traces of unkilld stock also assists very materially in the development of the texture desired in the finished product.

99. Settling the Soap.—"Fitting" (the term employed in English practice), "finishing," or "settling" the soap consists essentially in thinning the soap to the desired consistency with weak lye or water. The latter may be added during boiling or it may be derived from the steam condensed during this change. • The strengthening lye from the preceding change is withdrawn and the soap boiled up with live steam. Water is added in small quantities at a time until the soap is closed. The open steam valve is partially closed.

The boiling preparatory to settling requires but a comparatively short time, and the change may be finished early in the forenoon. Some free alkali is retained mechanically by the soap after the withdrawal of the strengthening lye.

This amount suffices to impart a slight sharpness or alkalinity to the settled soap. It is claimed by some that the soap should be settled in a perfectly neutral condition; others claim that the best results are obtained with a slight sharpness present.

100. The soap is boiled up to the top of the kettle and is then allowed to stand for about 5 days, during which time the contents of the kettle, in virtue of the different specific gravities, resolve themselves roughly into two portions, viz., the finished soap carrying about 30 per cent. of water, this proportion, however, varying with the grain of the finish, and the niger, which carries considerably more water than does the supernatant soap, as well as the impurities and coloring matter settled from it. The settling change is a very important one, for on its proper operation depends the success or failure of the boil of soap.

101. Niger.—The niger is the liquor from the settled soap, which contains the impurities and constitutes from 20 to 25 per cent. of the volume of settled soap in the kettle. • This proportion varies with the degree of hydration of the finished soap, the length of time allowed for settling, and the temperature of the mass. The more thinly the soap is finished, the larger will be the niger and the impurities of

the finished soap, other things being equal, will be more completely settled into it. Care must be taken not to finish the soap too thinly, for the excessive quantity of water added to the kettle not only makes the finished soap softer, but leaves a large bulk of niger to receive subsequent treatment.

On the other hand, a soap finished too closely, from the addition of an insufficient quantity of water, will separate the impurities very incompletely and yield a small niger. Other conditions being satisfactory, the longer the settling period, the more complete will be the separation of impurities with the formation of a niger of proper volume. If during this period the soap is allowed to cool too quickly, which is often the case with a small kettle or with one exposed to the weather, especially in winter, separation of the impurities is checked by a local or general cooling of the mass. The niger will then remain distributed unequally throughout the soap.

102. The settling change is not primarily a purifying change, but provides first for the production of a mass of a certain appearance and consistency, and when this is obtained and the proper conditions noted above are observed, the soap mass separates the impurities, thus forming the niger.

The soap mass is said to be finished "fine" or "coarse," or, respectively, "hard" or "soft," according to its degree of hydration. The condition described as "fine" or "hard" obtains when the soap taken up on the paddle and held in a slanting position falls from it in short, small flakes and cools rather quickly. The condition described as "coarse" or "soft" obtains when the soap falls from the paddle in large flakes and cools less quickly. The appearance and consistency must be learned by actual experience, by personal handling of the material itself.

103. Filling of Soap. — The detergency of soap is greatly increased by the addition of certain substances in aqueous solution, while the soap is in a fluid state.

Borax and sodium carbonate are employed, but the use of the former for this purpose is, as a rule, displaced by the cheaper alkaline carbonate.

As all surface water contains mineral salts, chiefly the carbonates and sulphates of lime and magnesium, it is necessary to neutralize these before the soap can exert any cleansing action. Insoluble soaps of lime and magnesium are formed from the combination of soluble soaps and the salts above mentioned, which impart the so-called hardness to surface water.

Sodium carbonate is a valuable addition to a soap, as a more economical use of soap is effected by its incorporation, as will be described subsequently, whereby the carbonate effects the neutralization of mineral salts, thus leaving the soap to exert its legitimate cleansing action. The value of sodium carbonate to increase the detergency of soap was early recognized, as was also its property of hardening the soap, thus permitting the incorporation of more water than would otherwise be possible without its use.

Sodium silicate lends itself readily to incorporation with fluid soap, as it possesses but little detergent power. It is primarily a cheapener. It imparts a smooth appearance to the finished soap, which on drying becomes extremely hard.

104. Sodium carbonate and sodium silicate are the chief filling agents. Their use in soap in considerable or small amounts is determined by the grade desired to be made and gives rise, respectively, to the terms *heavily filled* and *lightly filled*.

Mineral soap stock is a residuum from petroleum distillation that is frequently used in heavily filled soap. It imparts a smooth appearance to such soap, and thereby counteracts the tendency of the mineral salts mentioned to grain the soap when added in large amount.

The limit to the use of filling agents is determined by the intelligence of the consumer. The incorporation in soap of filling agents is not, strictly speaking, an adulteration, so

long as the product is sold at a price commensurate with its quality.

105. Crutching the Soap.—For the incorporation of filling material into soap, the belt-driven **crutcher** is generally employed. There are three styles of this machine, each possessing their several points of excellence. Style A, shown in Fig. 11, consists of a cylindrical vessel *a*, in which

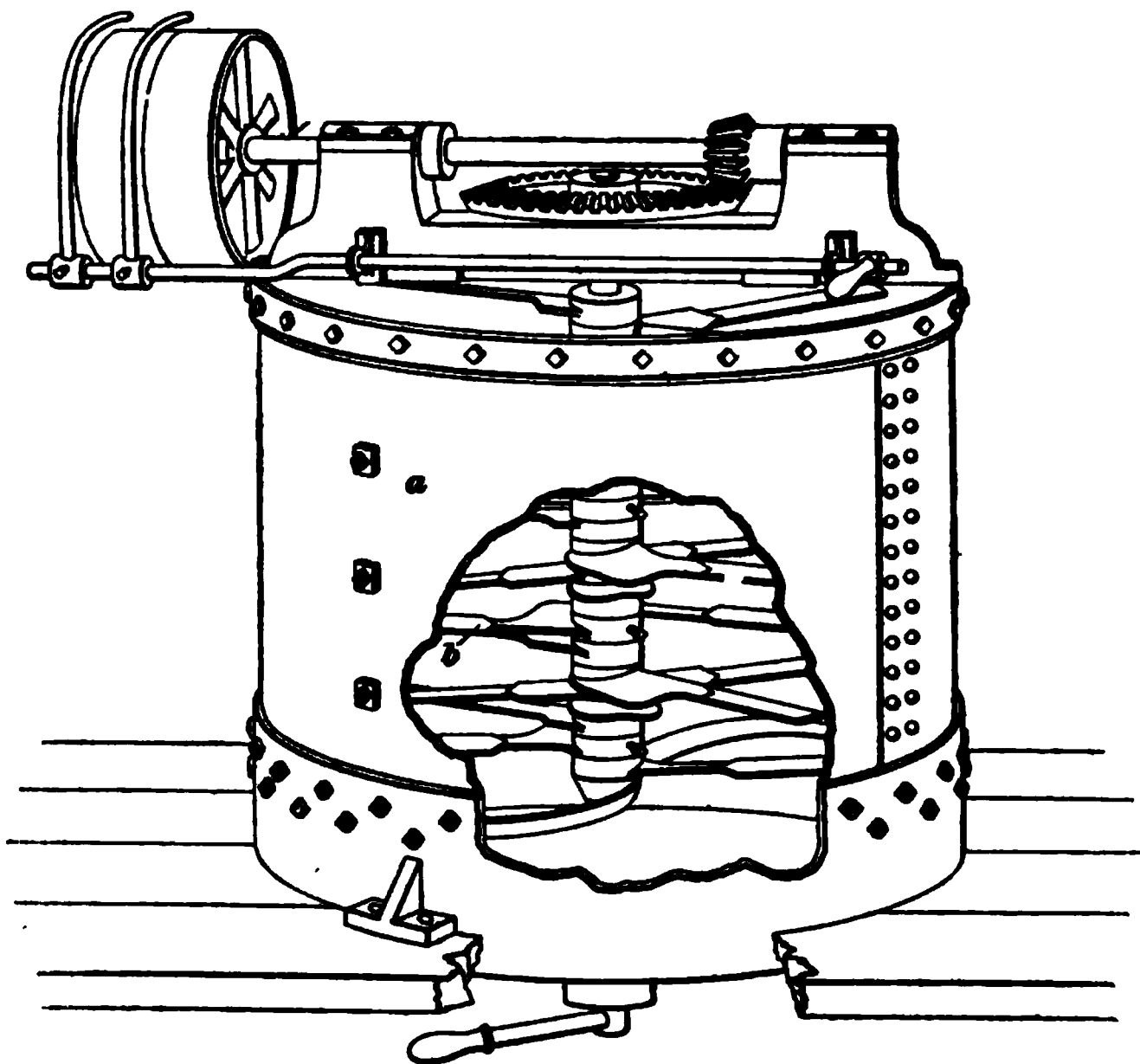


FIG. 11

is mounted a vertical shaft carrying a series of horizontal paddles *b*. These paddles rotate entirely within the body of the soap, which remains practically stationary. The blades of the horizontal paddles may be so constructed, however, as to impart an upward movement to the soap. ¶

With a crutcher constructed as shown in Fig. 11, it is impossible to incorporate air into the mass of soap, as the mixing is done entirely within the body of the soap.

106. The distinguishing feature of the crutcher, style B, shown in Fig. 12, is an inner concentric cylinder *a*, enclosing a broad Archimedean screw *d* mounted on a vertical

FIG. 12

shaft. Both the outer shell *c* and the concentric cylinder *a* may be steam-jacketed. This style is more commonly used without the steam jacket.

With this crutcher the entire mass of soap may be moved from below upwards through the central cylinder by means of the screw *d*, and downwards between the outer shell *c* and the concentric cylinder *a*, or in the reverse direction, according as the belt is advanced or reversed. By filling the crutcher within 2 inches of the top of the concentric cylinder *a* and rotating the screw *d* rapidly, much air can be incorporated into the soap, which is done in the manufacture of floating soap.

107. Style C, shown in Fig. 13, is essentially the same as style A. The shaft in this style is horizontal, with blades of varying length mounted in a screw-like fashion upon it. Soap

is pumped into the hopper *a* until the paddles *c* are covered to a depth of 2 inches. The crutcher is then started and run until the soap is crutched. The screw-like motion of the paddles works the contents of the crutcher towards the outlet *b*, which is closed by the counterpoise *d*. If the soap is not too thick, it will flow freely from the outlet *b*. If too thick, it is necessary to start the crutcher.

FIG. 12

Each type of crutcher may be steam-jacketed if desired. Where no separate remelter is part of the factory equipment, this feature is essential. The steam-jacketed crutcher can then serve as a remelter. The capacity of a crutcher is that of one frame, viz., 1,200 pounds.

108. Soap Pumps.—The liquid soap is transferred to the crutcher by means of a pump. The pumps used vary somewhat in construction and some of the most generally used will be described.

109. In the *Tabor rotary pump*, the working parts of which are shown in Fig. 14, *a* is the shell or case of the pump; *b* is the head covering the end of the shell; *c* is the piston that carries the valves; *d, d'* are the valves that pass through the piston in the ways *e, e'*, and as the piston revolves pass in and out, back and forth through the piston, following the inside lines of the shell; *f, f'* are the ways in the piston for the valves. The

FIG 14

piston *c* sets tightly against the shell at the point *f*, between the point of suction and discharge. Rotating the piston creates a vacuum in the suction pipe and the pump is set in operation.

110. The *Herscy rotary pump*, shown in Fig. 15, consists essentially of a cone-shaped casting *d*, carrying blades, the whole being mounted on the end of a piston shown at *e*. This piston rotates in a specially shaped case. When the piston rotates in the direction indicated by the arrow, suction takes place at the orifice *a* and discharge at *c*. The reverse occurs when the piston is rotated in the opposite direction.

111. The *Johnson rotary pump*, shown in Fig. 16, consists of an outside shell *a*, with suitable parts *b* and *c* for connecting suction and discharge pipes; two side plates, one of which is shown at *d*, with the cam *g* and inside of all a circular casting or piston *e*, in which are held the piston heads *f* operated by the cam *g*. As the piston revolves, the cam *g*

causes the piston heads *f* to move in and out of their ways, being at their extreme positions as shown in the figure. The block *h* separates the suction and discharge chambers.

112. The rotary type of pump is adapted for a variety of uses in the soap factory. Its simple construction, ease of operation, and freedom from expensive repairs give it many advantages over the ordinary steam pump. The chief uses



FIG. 15

for a pump are to transfer stock and caustic lye to the soap kettle, the finished soap to the crutcher, and waste-soap lye to the glycerine refinery. On the discharge side of the pump there should be connected a steam pipe to blow out and clean the interior of the pump and the discharge pipe after use.* As shown in Figs. 14 and 15, the pumps are provided with tight and loose pulleys, upon the latter of which the belt may be shifted when the pump is not in use.

For pumping the soap from the kettle to the crutcher, the pump should be placed below the level of the draw-off pipe in the kettle and the outlet or discharge pipe should be of slightly smaller diameter than the intake pipe. In this way



FIG. 16

gravity aids the feed and provides for a steady discharge. The rotary belt-driven pump represents a great improvement in productive economy over the old-time ladle and bucket as an instrument for transferring soap from vessel to vessel.

MANUFACTURE OF SOAP

(PART 2)

FILLING MATERIALS AND THEIR PREPARATION

1. Soda Ash.—Soda ash is the most valuable addition to laundry soap. It is introduced in the crutcher in the form of a saturated solution. The crutcher room is usually provided with a steam-heated kettle with an agitator for dissolving the soda ash as required. During the afternoon of the day previous to crutching, a quantity of solution sufficient for the entire boil of soap is made up. With a sal-soda kettle of given capacity, the quantity of soda ash and water necessary to produce a solution of the required density is easily ascertained by experience. The mixture is boiled with open steam to a density of 31° to 32° Baumé while hot. During the night the impurities in the solution, introduced by the soda ash, will have settled to the bottom and the solution cooled to a temperature of about 155° F. and with the specific gravity increased to 34° Baumé.

The draw-off pipe is best placed from 2 to 3 inches above the bottom of the sal-soda kettle to avoid the removal of the settled impurities. The kettle, or tank, is best placed on an elevated platform, so that the liquor can flow, by gravity, into the crutcher in regulated quantities as desired. A 34° Baumé solution contains 27.97 per cent. of anhydrous sodium carbonate; 100 pounds of the solution will therefore contain 27.97, or practically 28, pounds of the dry salt.

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TABLE I
SPECIFIC GRAVITIES OF SOLUTIONS OF SODIUM CAR-
BONATE (LUNGE). (A) AT 15° C.

Specific Gravity	Baumé	Twaddell	$\%Na_2CO_3$	$\%Na_2CO_3$, 10 Aq.	1 Cbm. Contains Kg.	
					Na_2CO_3	Na_2CO_3 , 10 Aq.
1.007	1	1.4	0.67	1.897	6.8	18.2
1.014	2	2.8	1.33	3.587	13.5	36.4
1.022	3	4.4	2.09	5.637	21.4	57.6
1.029	4	5.8	2.76	7.444	28.4	76.6
1.036	5	7.2	3.43	9.251	35.5	95.8
1.045	6	9.0	4.29	11.570	44.8	120.9
1.052	7	10.4	4.94	13.323	52.0	140.2
1.060	8	12.0	5.71	15.400	60.5	163.2
1.067	9	13.4	6.37	17.180	68.0	183.3
1.075	10	15.0	7.12	19.203	76.5	206.4
1.083	11	16.6	7.88	21.252	85.3	230.2
1.091	12	18.2	8.62	23.248	94.0	253.6
1.100	13	20.0	9.43	25.432	103.7	279.8
1.108	14	21.6	10.19	27.482	112.9	304.5
1.116	15	23.2	10.95	29.532	122.2	329.6
1.125	16	25.0	11.81	31.851	132.9	358.3
1.134	17	26.8	12.61	34.009	143.0	385.7

(B) AT 30° C.

Specific Gravity	Baumé	$\%Na_2CO_3$	$\%Na_2CO_3$, 10 Aq.	1 Liter Contains Grams	
				Na_2CO_3	Na_2CO_3 , 10 Aq.
1.308	34	27.97	75.48	365.9	987.4
1.297	33	27.06	73.02	351.0	947.1
1.285	32	26.04	70.28	334.6	902.8
1.274	31	25.11	67.76	319.9	863.2
1.263	30	24.18	65.24	305.4	824.1
1.252	29	23.25	62.73	291.1	785.4
1.241	28	22.29	60.15	276.6	746.3
1.231	27	21.42	57.80	263.7	711.5
1.220	26	20.47	55.29	249.7	673.8
1.210	25	19.61	52.91	237.3	640.3
1.200	24	18.76	50.62	225.1	607.4
1.190	23	17.90	48.31	214.0	577.5
1.180	22	17.04	45.97	201.1	542.6
1.171	21	16.27	43.89	190.5	514.0
1.162	20	15.49	41.79	180.0	485.7
1.152	19	14.64	39.51	168.7	455.2
1.142	18	13.79	37.21	157.5	425.0

2. The quantitative properties of sodium-carbonate solutions above a density of 18° Baumé are determined at a temperature of 30° C. At a temperature of 15° C., solutions of the density shown in Table I (B) will crystallize, forming the hydrated salt $Na_2CO_3 \cdot 10H_2O$, commonly known as **sal soda**.

3. Concentrated solutions of sodium carbonate, when added in considerable amounts to any soap and particularly to soap made from soft stock, which we have learned sweats more readily than soap made from firmer stock, will soon cause the soap to effloresce. This is a most disagreeable property and detracts greatly from the appearance of the product. Also when added in greater quantity than the soap can assimilate, the soda ash tends to grain the soap. With heavily filled cheap soaps, this condition represents the limit of the addition; soda ash and other filling agents possessing detergent properties then become adulterants.

4. **Borax.**—Borax is a mild alkali and is a desirable addition to soap; it possesses all the advantages of soda ash with none of its caustic properties. Owing to the higher cost, its use in soap manufacture is limited; with certain brands, sufficient only is used to justify in a measure the title given to the soap.

5. **Sodium Silicate.**—Sodium silicate, commonly called *water glass* or *soluble glass*, next to soda ash, is the most extensively used filling agent.

Its consistency lends itself to ready incorporation with the semi-fluid soap. It possesses detergent power, and when used with due regard to the price of the finished soap, it is a valuable addition to the ordinary household soap.

It is made by fusing pure white sand and soda ash in suitable proportions in a reverberatory furnace. The resulting glass is broken into fragments and introduced into a digester, already charged with an amount of water to yield a solution of any density desired, and high-pressure steam admitted. When solution is complete, the contents of the digester are

run into barrels, in which shape it is received by the soap maker. Sodium silicate is generally used at a density of 40° Baumé, in which condition it has the consistency of thick molasses, is transparent, and hardens quickly on exposure. At ordinary temperatures it will flow readily from the barrel. It must be in a fluid condition when used. In cold weather it may be softened by blowing steam into the barrel. If the head of the barrel be removed and the contents exposed to the air, those varieties deficient in alkaline strength will "jelly" or separate free silicic acid through the displacement of the latter acid by the stronger carbonic acid of the atmosphere. This may be overcome by stirring up the mass with strong caustic-soda lye, whereby a combination of the silicic acid with alkali is effected.

6. When intended for soap-maker's use, the sodium silicate in solution contains approximately 75 per cent. of SiO_2 and 25 per cent. of Na_2O . The more silicic acid the product contains, the more difficultly soluble it is in water. The composition of commercial sodium silicate, as it occurs in solutions of different densities to be used for different purposes is variable, depending on the proportions of soda ash and silica used in the original charge and on the amount of water in which the fused mass is dissolved in the digester. There is practically no "neutral" silicate; that which is called "neutral" contains an excess of silicic acid, although it is not acid to litmus.

7. On aging, soap filled with sodium silicate becomes very hard. When well mixed with the soap, a reasonable proportion greatly improves the appearance and enables a larger quantity of sal soda to be carried without the efflorescence that would soon be produced without its use.

While sodium silicate possesses detergent properties, it is used primarily as a cheapener. By the use of suitable proportions of solutions of soda ash and sodium silicate, the quality of laundry soap is greatly improved. The soap is made more durable from the hardness produced by the

crystallization of the salts; also the rapid drying of the soap is retarded.

When used in excess in cheap soap, they not only permit the absorption of a larger amount of water than would otherwise be practicable, but also, when used in soap made from soft stock, they impart a firmness which, without their use, would not be possible, without having previously transformed the product into a variety of boiled-down soaps.

8. Other Fillers and Adulterants.—Ground quartz, or silica, marble dust, mineral-soap stock, a petroleum residuum, starch, and talc may be mentioned as fillers. They are adulterants pure and simple.

9. Perfuming the Soap.—With the best grades of laundry soap it is common practice to add a small amount of either a single essential oil or a mixture of them in order to produce a pleasant and lasting perfume. The quantity added is seldom in excess of $2\frac{1}{2}$ pounds per frame. The selection of the oil, or mixture of oils, is best determined by experiment, the object being to choose an oil, or mixture of oils, that will best counteract the resinous odor inseparable from rosined soaps.

The perfuming of soap, both domestic and laundry, will be discussed at length later.

10. Crutching the Filler and Perfume Into the Soap.—During the settling period previously described, the soap will have cooled from that temperature at which it was when in contact with steam to about 160° F. The temperature best adapted to crutching depends largely on the character of the stock used in the soap and on the quantity and temperature of the filling material added in the crutcher. The temperature of crutching need not be as high for soap made from soft stock, because of its natural softness, as for soap made from firm stock. Also in the summer a temperature 10° lower than that in the winter is permissible.

If the crutcher is provided with a steam jacket, the soap can be easily kept at any desired temperature. Such devices entail the use of a large amount of steam, and moreover

for this purpose are not absolutely essential. It is best to add the soda-ash solution at the same temperature as that of the soap, and when prepared as has been explained, it is at the proper temperature when the soap is ready to crutch.

11. When ready to crutch, the swing-joint pipe is lowered a short distance below the level of the soap in the kettle and held in place by a chain, as shown in Figs. 8 and 9, *Manufacture of Soap*, Part 1. The rotary pump is started and the crutcher filled.

The speed of crutching is best determined by experience. For a soap of the character we have been discussing, 80 pounds of sal soda are run in and the mass crutched until fairly homogeneous. The sodium silicate is then added and crutched in. The perfume is added last and the soap crutched until a portion removed on a paddle is perfectly homogeneous in texture and smooth in appearance.

Under satisfactory conditions, from 3 to 5 minutes is sufficient to crutch a frame of soap. With completely saponified soap, pumped to the crutcher at a temperature not exceeding 160° F. in winter, and carrying a reasonable amount of filling, no trouble need be experienced in crutching. The primary conditions for satisfactory work are that the stock shall be completely saponified and the soap well settled. As to the soap being neutral or having a slight sharpness, there is a difference of opinion.

12. At the end of the crutching period, the outlet in the bottom of the crutcher is opened and the contents emptied into a frame standing on the floor immediately below the crutcher. Where it is possible to operate two crutchers, mounted as shown in Fig. 1, the work is greatly facilitated, as the crutching can proceed in one while the other is being emptied, and vice versa.

The swing-joint pipe in the soap kettle is gradually lowered as the crutching proceeds until the niger is reached. The attendant ascertains this in advance by tossing up the soap with a long paddle, thus disclosing the dark niger below the level of the lighter colored soap.

We have been discussing a kettle charge of 22,500 pounds of glyceride stock to which one-third its weight, or 7,500 pounds, of rosin have been added. The yield for all practical purposes is 50 per cent. We have therefore in the kettle previous to crutching, 45,000 pounds of finished soap. Considering approximately one-fourth of the total volume of soap for the niger, this kettle charge should yield us about 35 frames of soap.

FIG. 1

In the crutcher there have been added 80 pounds of soda-ash solution, 20 pounds of silicate of soda, and $\frac{1}{4}$ pound of perfume, or approximately 8 per cent. of filling to each frame. This increase with the material used is the best practicable maximum, although it may be increased very considerably by the use of adulterants—viz., mineral soap stock, starch, or talc.

13. The crutching of the soap from a 35-frame kettle, if begun promptly in the morning, should be finished by 11 o'clock. After the last frame of soap, free from contamination of niger, has been pumped from the kettle, the pipe

leading from the kettle to the crutcher should be blown out with live steam. The discharge should flow into the kettle.

The swing-joint pipe is now pulled up and secured to the side of the kettle and steam turned on. The strengthening lye from another boil is run in and the mixture well boiled through. During this time grease is added slowly, and the caustic alkali present in the niger and the strengthening lye is exhausted in the same manner as the stock is killed on the stock change. In this case, however, the soap stock is added to the caustic soda already in the kettle. When the alkali has been completely absorbed, the contents of the kettle is boiled up and grained as described in *Manufacture of Soap*, Part 1. After settling over night, the niger lye is withdrawn and a new boil begun on the grained niger.

By referring to the "Chronology of a Boil of Settled Rosin Soap" in Table XIV, *Manufacture of Soap*, Part 1, we see that on the twelfth day from the beginning of the boil the kettle is available for a new boil of soap. The distribution of the work over 12 days, as described, is variable, depending on a multitude of conditions, which are different in different factories. The time may be considerably lessened, if the demand for soap warrants it, by continuing the work at night and allowing less time for the settling periods.

14. Framing the Soap.—The soap frame, shown in Fig. 2, has attained its present shape and size as the result of experience in handling soap in this stage of its manufacture. It consists essentially of an uncovered box, of a capacity of from 1,000 to 1,200 pounds of soap, with removable sheet-steel sides, and with ends of wood or sheet steel set on a wooden bottom, which is mounted on truck wheels.

There are various styles manufactured, all with the single aim of producing a box that can be readily put together and taken apart, that will be durable, as light in weight as is consistent with strength, that can be easily moved about, and that will possess the most important qualification—viz., tightness. The dimensions of the frame are variable and are determined, especially the width, by the

size of the bar into which the slabbed soap is cut, it being the aim to reduce the percentage of scrap at the cutting table as low as possible.

In the frame-room equipment, it is best to have an extra bottom for each frame. The sides of a frame of soap may be removed after the soap has cooled sufficiently to

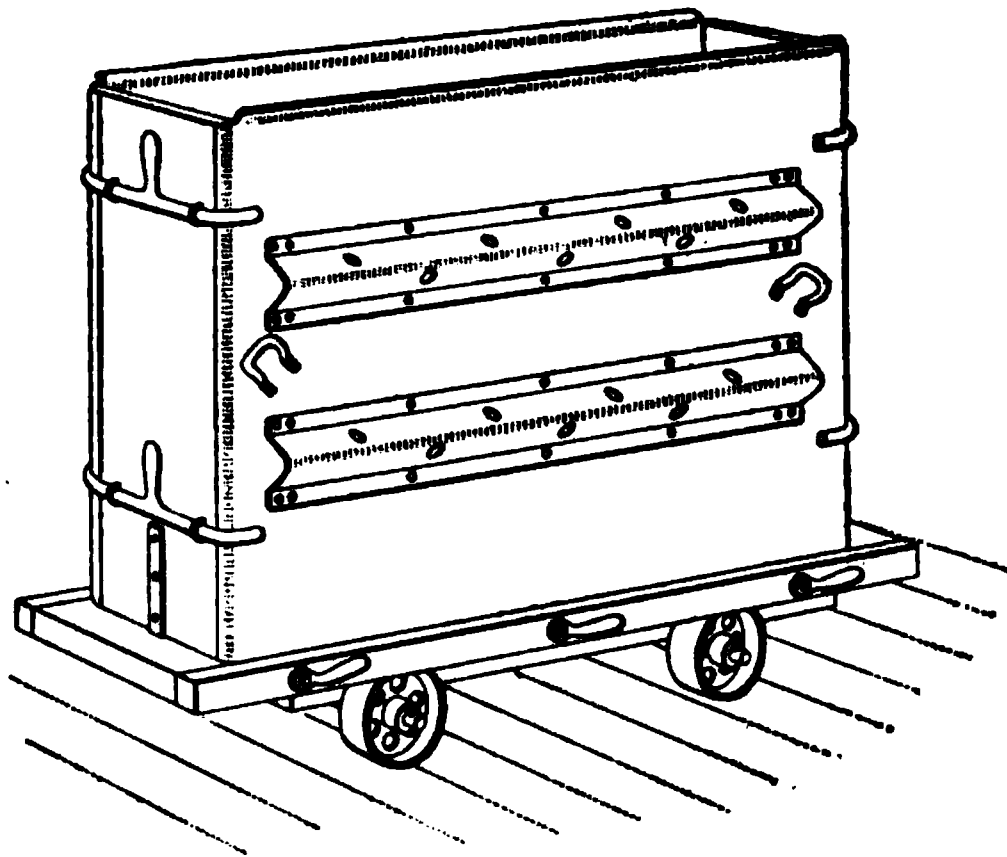


FIG. 2

permit it and the sides mounted on the extra bottom. This is a most economical method, the usefulness of the frame being doubled, as the original bottom carries the soap through the slabber and to the cutting table, by which time the original sides may be used to enclose a new framing of soap.

15. Previous to crutching, or as it proceeds, a sufficient number of frames are put in readiness. Where the crutchers are operated in pairs, an empty frame should stand ready to receive its charge as the preceding one is being filled. The filled frame, with the soap smoothed down with a short paddle into the edges of the frame and heaped up longitudinally in the middle, is pushed into its stand in the frame room. Here it is allowed to remain for a varying period, usually from 3 to 5 days, which period is determined by the demands of the factory, the season, the character of the soap, and the temperature at which it was framed.

Before the advent of the steam-driven crutcher, the soap was laboriously crutched in the frame with a hand crutch.

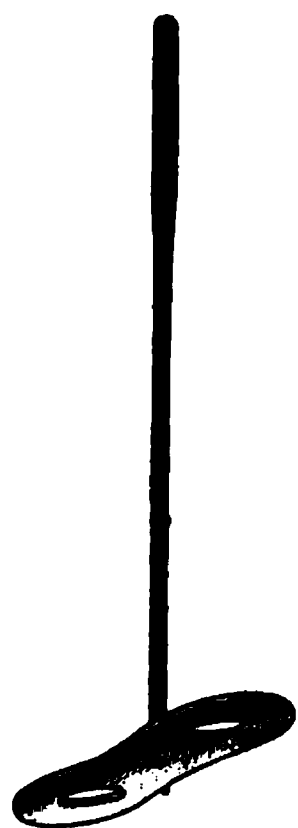


FIG. 3

The hand crutch, shown in Fig. 3, is still in use for soap that may be crutched at too high a temperature to prevent the settling of the filling material to the bottom of the frame; to distribute the material used to produce a mottle; or to distribute the reduction of temperature uniformly throughout the entire mass as cooling proceeds, thus preventing the separation of the softer from the harder soap.

With soap made of the ingredients given in *Manufacture of Soap*, Part 1, Art. 84, and filled as we have noted, the sides may be removed from the frame on the second day after crutching. After scraping the sides of the soap, after removing the frame sides, to remove stains and blotches of adhering matter, if present, the soap is ready to be slabbed.

16. The Slabbing Machine, or Slabber.—This device varies greatly in simplicity and cheapness of mechanical construction. It is a development of the old hand method of drawing a wire in parallel lines longitudinally through the mass of soap. In the machine slabber, be it hand or steam-driven, the direction of application of the power is reversed, the frame of soap being forced through a series of parallel wires arranged on a framework to the height of the soap to be slabbed. The essential mechanical features of the machine slabber are shown in Fig. 4.

Piano wire is the best for soap cutting. It is mounted on the frame head *a* in parallel rows at a distance apart corresponding to the width or height of the unpressed bars of soap. The frame of uncut soap is shown at *b*. By means of a key set in the sides of the frame head, the wires may be tightened or loosened as required. With soap that has stood for some time, it is often necessary to remove, for a short distance on both ends of the frame, the layer of hard

soap. This enables the wires to enter the soap easily, without undue stretching and frequently breaking, and to separate



FIG. 4

from it without the abruptness that so often injures the wires.

17. Cutting the Soap.—From the slabber the soap is moved to the cutting table, where it is cut into bars. The fundamental principle of all cutting tables is shown in Fig. 5. A slab is lifted from the pile *a* lying on the frame bottom and transferred to the table *b*, whence it is pushed lengthwise through one or two wires held in the cutting head *c*, whereby it is cut into two or three narrower slabs as wide as the single bar of soap is long. These slabs are then cut at right angles by another attendant to dimensions corresponding to the width and thickness of a single bar. The last cutting movement pushes the slabs through the wires in the cutting head *d*, whence as single bars they are pushed on to a rack supported by an extension of the cutting table. Here, by a slight upward and horizontal motion, the individual bars are separated that air may circulate between them. The separate racks are placed upon a truck until its full

quota has been received. The truck load is then pulled into the drying room.

FIG. 5

18. The slabbing and cutting of soap are purely mechanical processes and have for their object the division of the frame of soap into bars. The greatest care to be exercised at these stages is to slab and cut the soap to such dimensions that the amount of trimmings, or scrap, is reduced to a minimum.

Freshly cut soap is soft, sticky, and opaque, and if properly crutched should be homogeneous. It contains from 30 to 35 per cent. of water, according to the manner in which the soap was settled and the nature of the additions during crutching. In calculation, 100 parts of glyceride soap stock are considered to yield 150 parts of finished soap. The yield is less with rosin alone, and with some stock it may be as great as 57 per cent.

An analysis of pure settled soap at this stage presents the following data:

Fatty anhydrides.....	61.80%
Combined alkali.....	7.21%
Anhydrous soap.....	69.01%
Water.....	30.99%
Total.....	100.00%

19. Drying the Soap.—Aside from the processes in the kettle, there is no stage in the manufacture of settled soap that requires greater care in its operation than the treatment received in the drying room. Improperly dried soap, while it may cause no great trouble in the press, is subject to rapid deterioration in appearance, and the influence of this one factor on its ultimate distribution demands that its final treatment in the factory be the subject of the closest attention of the soap manufacturer.

Previous to the introduction of the rapid-drying apparatus, the moisture in the exterior parts of the bar was allowed to evaporate spontaneously. By this method the drying of soap was an extremely slow and unsatisfactory process, depending largely on atmospheric conditions. A stove in a closed room was a great improvement. This primitive method, with its manifest disadvantages, was succeeded by a system of hot-air circulation by natural draft. Steam heat by simple radiation from pipes was also employed, and with the introduction of forced draft the elements of the system of drying in use at present were established.

20. Purpose of Drying Room.—The purpose of the drying room is to hasten the evaporation of water from the surface of the bar, so that there may be formed a thin crust of comparatively hard soap, which serves to retard further evaporation from the interior of the bar, and which allows the bar to be pressed and stamped without the soap adhering to the dies. Without the formation of this skin of firm soap, the latter process is very unsatisfactory.

On cutting a bar of soap into halves, this superficial drying becomes plainly evident. The soap when removed from the drying room and after being pressed has a smooth, glossy,

and translucent surface, which condition is in marked contrast to that observed at the cutting table. During the drying process, from 3 to 5 per cent. of water has been expelled entirely from the surface of the bar, while the interior contains the amount of water originally present, viz., 30 to 35 per cent. We have in this unequal equilibrium of moisture contents between the exterior and interior parts of the bar a partial explanation of the sweating to which settled soaps are universally susceptible.

This accumulation of moisture does not develop until after the bar is wrapped and packed. If we should take a freshly pressed bar of soap and allow it to remain exposed to the atmosphere, it would dry but would not sweat, assuming, of course, that the atmosphere has not attained the dew point. The moisture in the interior of the bar has passed through the hard surface into the atmosphere, and this process will continue until an equilibrium of moisture contents throughout the bar has been attained.

21. With soap wrapped and packed in a box, the conditions are naturally entirely different. The tendency for the moisture to pass from the interior of the bar to the drier surface remains, but further evaporation from the exterior of the bar is checked. Here the moisture accumulates and softens the soap, which in turn adheres to the wrapper. If the soap contains an excessive quantity of mineral salts, these are carried in an aqueous solution to the surface and on subsequent evaporation of the water form an incrustation. Boxed soap in storage should not be subject to unnatural fluctuations of temperature.

22. Drying Soap by Forced Ventilation. — The mechanics of the modern soap-drying room represent more the adoption of a similar process employed in other departments of industry than it does a natural evolution from previous efforts in this particular field. The use of centrifugal fans in the production of artificial draft dates from the 16th century, but it was not until Stevens's experiments in the early part of the 19th century that the devices for

artificial draft resolved themselves into the systems of ventilation we know to-day, viz., the *plenum* and *vacuum*, or, respectively, the *forced* and *induced*, draft. In connection with the drying of soap, ventilating fans may be divided into two general classes, viz., the centrifugal fan, or blower, and the propeller, or disk fan. The former is more generally confined to ventilation by forced draft and is designed



FIG. 6

primarily for removing air under pressure. Fans of the disk type, shown in Figs. 6 and 7, are not adapted to plenum ventilation, where it is desired to move large volumes of air quickly and under considerable resistance. They find an extensive use and are very satisfactory for moving air under slight resistance, as under conditions met with in the ventilation of soap-drying rooms.

23. Both systems of ventilation, the plenum and the vacuum, are used in the drying of soap. The equipment of a drying room under the plenum system comprises, as a rule, a disk fan, two arrangements of which are shown in Figs. 6 and 7. In the arrangement shown in Fig. 7, the fan is operated either by a belt from shafting or by a direct- or belt-connected engine and a sectional heater that consists of steam pipes enclosed in a sheet-iron case *a*, communicating with the discharge of the fan case *b*. Air may either be forced through the heater and discharged at the desired temperature into the drying room, or the fan

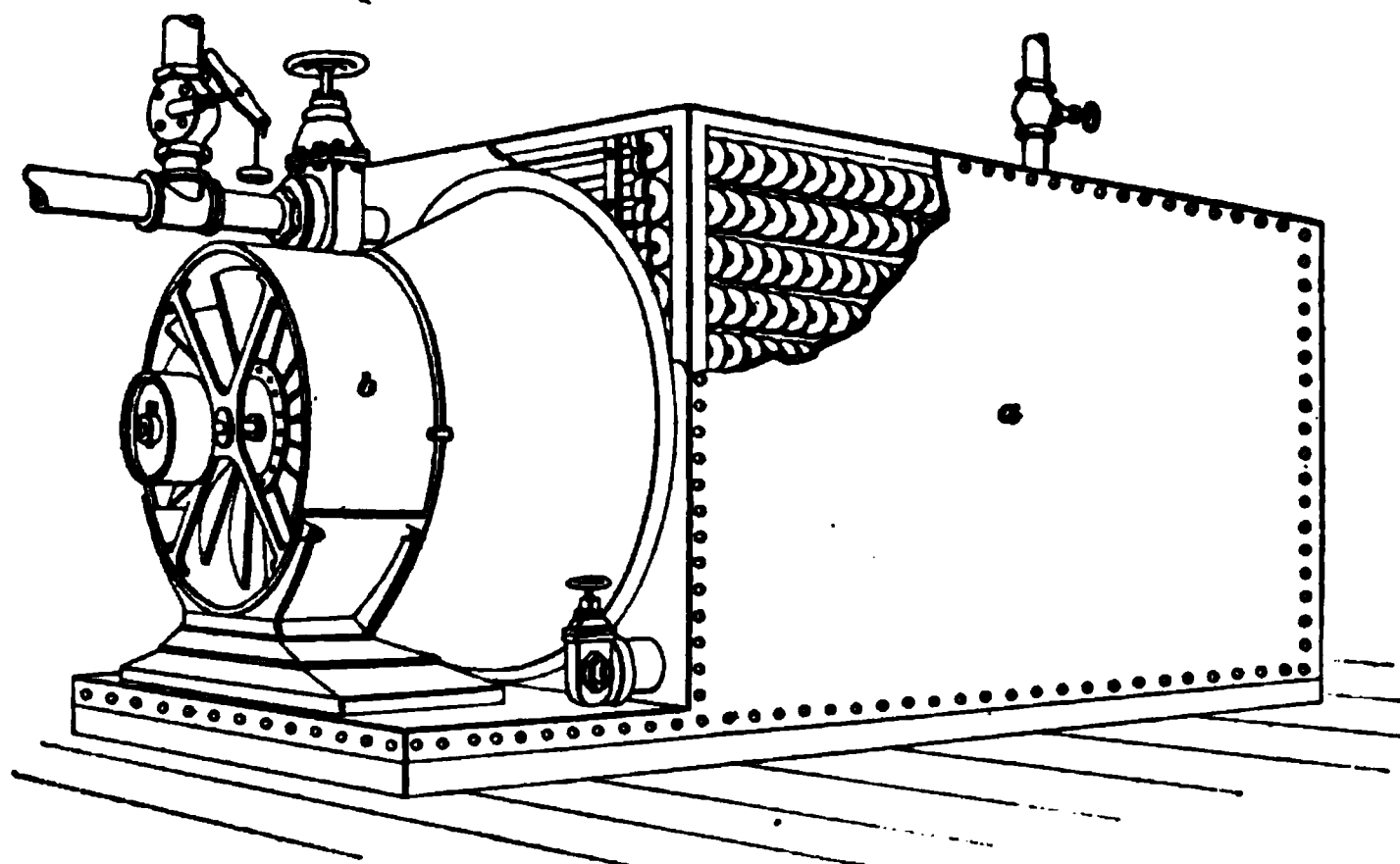


FIG. 7

may be interposed and air drawn through the heater and then discharged into the drying room. As the results produced are the same in either case, convenience of application will determine the arrangement.

With forced draft, the drying room is best constructed so that heated air enters at one end and leaves at the other, while freshly cut soap is introduced at the side, and as the drying progresses is withdrawn at the opposite side. The heater and fan may be placed at opposite ends of the room and the fan used to exhaust the warm and moisture-laden air. With this arrangement we have an example of the

vacuum system, or drying by induced draft. The combination of heater and fan shown in Fig. 7 is replaced with advantage by the location of the heating coils at one end of the drying room or arranging them in rows throughout the drying room between the trucks of soap. The cheaper and simpler disk fan set in the framework of the wall, as shown in Fig. 6, is, with this arrangement for the drying of soap, equally efficient and satisfactory. The hot-blast drying apparatus shown in Fig. 7, although compact, may occupy valuable space. The exhaust-steam connections with the necessary insulation are simple and easily made.

24. Heating of the Drying Room.—By distributing the pipes of the sectional heater throughout the drying room in rows parallel to the trucks of soap and under openings immediately above for the admission of cold air, not only is a greater uniformity of the drying process obtained, but the use of the cheaper disk fan, which is admirably adapted for ventilation by exhaustion, is permitted. Exhaust-steam connections are made through the floor, and by means of suitably placed valves exhaust steam may be cut off from any section, thus varying the capacity of the drying room at will.

In the drying process, air fulfils two functions: it carries to the moist soap heat necessary for the evaporation of the water and it serves as a vehicle for the removal of the vapor. The capacity of air for heat is very small, its specific heat being but .238, with water as 1. Its capacity for vapor depends directly on its temperature and its relation to the dew point, naturally diminishing as the point of saturation is reached. With a rise of temperature the capacity of air for moisture increases. It is estimated that air at 72° F. has a threefold greater capacity for aqueous vapor than the same volume at 42° F.; at 172° F., its capacity for vapor is more than eighty times as great. Increase of temperature thus means the more rapid formation of vapor, with a much greater increase in the capacity of air for absorbing it.

25. Requirements of the Drying Room.—The essential requirements of the heating and ventilating apparatus

of the drying room are that it provide a large volume of air at the required temperature and maintain it in rapid circulation. A comparatively low temperature, viz., 80° to 100° F., is productive of the best results. Air maintained at a temperature in excess of 100° F. for any considerable period causes the soap to undergo an appreciable softening, with the development of more or less discoloration.

The temperature at which soap will melt depends primarily on the nature of the stock from which it is made and the proportion of water present. In the drying of "green" soap, it is desirable that the currents of warm air should circulate lengthwise of the bar, in order that the

largest extent of evaporative surface may be exposed and the drying process thus hastened. The end of the drying period is easily learned by experience from the appearance of the bar of soap; it varies from 6 to 12 hours.

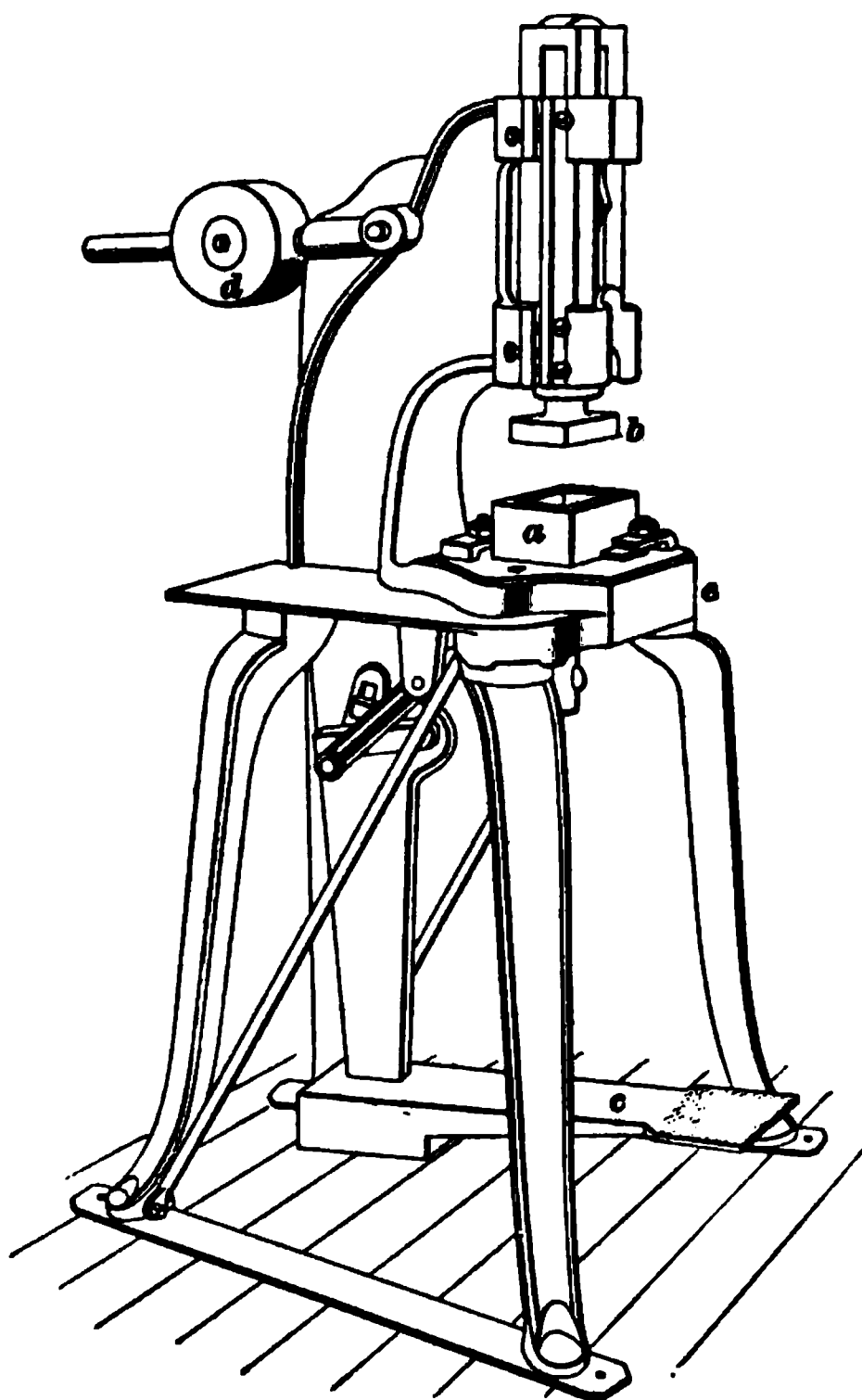


FIG. 8

26. Pressing of Soap.—Hard soap was originally sold in this country as cheese is sold to-day. Later the soap was cut into bars and packed, with the wrappers in bulk, in a box, the retailer himself wrapping the soap as sold. Another step brings us

to present conditions, in which the freshly cut bar is pressed, stamped, and wrapped.

In Fig. 8 are shown the essential features of a foot-power soap press. It enables the operator to deliver a sudden blow to the cake of soap placed with the *thumb and forefinger* of the right hand in the die box *a*. An operator on a press of this kind will press 45 bars per minute. To prevent soap from adhering to the die *b*, the cake is placed lightly on a bunch of waste saturated with brine or a mixture of vinegar and water, placed to the right of the die box *a*. After the blow has been delivered by the right foot placed on the lever *c*, the upper die *b* is lifted by the counterpoise *d*, which also serves to add momentum to the force of the blow, a lever action lifts the pressed bar out of the die box *a*, whence it is transferred, by the left hand of the operator, to a table placed at his left. The foot-power soap press is built in a number of styles, some being partly operated by steam, as shown in Fig. 9.

27. Steam Foot-Press.—This apparatus is provided with a steam cylinder *a*, the piston *b* of which is directly connected with the lever *c*, which controls the dies *d* and *e*. A system of valves is arranged at *f* and *g*, from where, by the slight pressure of the foot upon the treadle *h*, steam is admitted to the cylinder *a* and a quick, powerful blow given to the soap placed over the die box, the lower die *e* of which is elevated as shown in Fig. 9. The lever returns instantly, withdrawing the upper die and elevating the lower one, from which the cake of soap is removed by the left hand of the operator. With rapid work, upwards of 1,500 cakes per hour can be pressed on a machine of this type.

28. Automatic Steam-Power Soap Press.—The belt-driven, automatic soap press is a direct development of the old foot-power press, and arose from a demand for greater rapidity of operation. Several are on the market with a guaranteed capacity of from 60,000 to 75,000 cakes per day of 10 hours. They are perfectly automatic in their

action, require but little attention, and for a factory producing upwards of 400 boxes of soap per day, such a machine is a safe purchase.

The more improved forms admit of interchangeability of the dies, thus permitting the soap manufacturer to use any die that he may have in stock. Thus all the brands of a

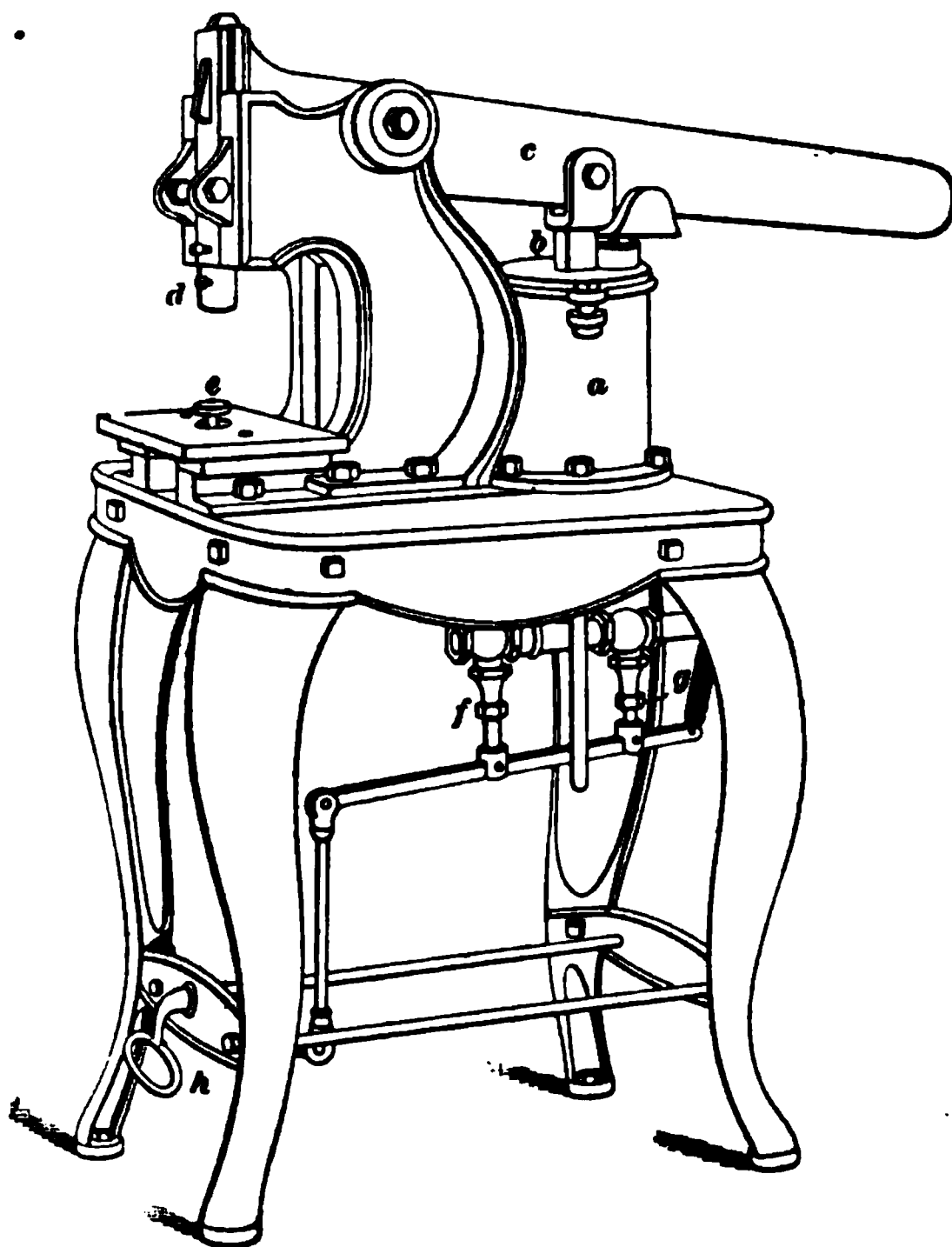


FIG. 9

single factory can be pressed on the same machine, with no longer time required to change the dies than on the old-style foot-press. In Fig. 10 is shown one of the latest forms of a soap press of this character. To describe it briefly, power is applied to the pulley *a*, which in turn operates the feeding belt, running on the surface of table *b*, which carries the cake of soap to the drop box *c*, down which it falls

between two dies impelled in a horizontal plane at *d*. The cake of soap as it falls down the drop box *c* is caught

FIG. 10

between the two dies, pressed, and dropped upon the belt *e*, which carries the pressed bar to the wrapping bench *f*, where it is wrapped and packed in boxes.

In Fig. 11 is shown what is called a "three-die rotary soap press." It consists essentially of double cylinders *a* and *b* placed end to end, in which rotate cams that automatically bring together and withdraw horizontally two dies that meet in their corresponding die box, of which three are arranged 120° apart. The dried soap is placed from the racks on to the feeding belt *e*, from which each bar is singly moved forwards to the die by a "finger" on the chain belt

FIG. 11

passing over the pulley *d*, to which power is applied at *c*. A set of dies is shown at *f*. The pressed soap is dropped on the belt running in the box *g*, which carries it to the wrapping bench.

Among the many advantages possessed by automatic soap presses are chiefly the perfect work, capable, at a maximum speed, of 60 bars per minute; the regulation of the pressure to suit the character of the soap, and the insurance of safety to the attendant from loss of fingers. The automatic

power press of the capacity stated will do the work of five foot-presses with one-third the labor.

29. Soap Dies.—The kind of die used is determined by the kind of soap to be pressed. The function of a die is twofold: it conforms the yielding mass of soap to a definite shape and imprints upon it, in either elevated or sunken figures, or both, usually, the brand of the soap and the name of the manufacturer or vender. The earliest form is the hand stamp, shown in Fig. 12, by means of which the brand or maker's name is impressed upon the freshly cut bar.

FIG. 12

The second form is the box die shown in Fig. 13. In this form of die the mold feature is introduced in pressing the soap from above and below in an enclosed space called a *box*. The upper die *a* and lower die *b* fit closely to the interior of the box *c*, and are so adjusted with the press as to cause their, respectively, downward and upward movement at a separating distance corresponding to the thickness of the pressed bar, to which dimension the thickness of the unpressed bar closely approximates. There is thus insured such a distribution of the soap in the mold as to fill every part of it.

FIG. 13

The same box and dies may be used for different brands by means of detachable name plates. In fitting the box to the bedplate of the soap press, shown at *e* in Fig. 8, by means of the shanks shown at *d*, Fig. 13, care should be taken to insure a perfectly vertical motion of the upper and

lower dies, otherwise they will rub against the interior sides of the box, with the result that the accuracy of the die will soon be destroyed.

In the molding and stamping of milled soap, dies of a different construction must be used because of the greater firmness of soap of this character. A powerful and sudden blow must be delivered, the construction of the die expelling all surplus soap from the cavity, instead of, as in the more yielding laundry soap, being forced into every part of it. The pin, or shoulder, die is so called from the use of pins and sockets to guide the upper and lower dies, thus preserving their accuracy. The term shoulder has reference to the base supporting and receiving the four pegs. As shown in Fig. 14, the third form consists of two dies: (a) the top

(a)

FIG. 14

die and (b) the bottom die; without a box, each forming the face and one-half of the cake. The edges c and c' of the dies strike together, thus forcing out all surplus soap. The guide pins d , d' , d'' , and d''' of the top die serve to guide it squarely on the bottom die, the shoulder of the bottom die, receiving the guide pins in the corresponding holes d , d' , d'' and d''' , bear the force of the blow, and preserve the cutting edges c and c' . These dies are also made with removable panels or name plates, thus permitting a number of brands to be stamped with the same die by inserting a different brand, or name, plate in the base of the bottom die.

The fourth form is a combination of the pin, or shoulder, and box dies, and in addition to possessing the qualities of each, it admits of the pressing of cakes of different thicknesses

and weights in virtue of the removable lower die placed in the box. As shown in Fig. 15, *a* is the removable lower die made in varying heights to produce a bar of corresponding thickness. It fits into the box *b*, which is fastened by the

FIG. 15

shanks *c* and *c* to the bedplate of the presses shown in Figs. 8 and 9; *d* are the guide pins of the top die *e*, *f* are the shoulders of the lower die

The dies are made of gun-metal alloy or rolled brass. They should be substantially made with all moving parts carefully fitted. The engraving should be so executed, with figures of uniform bevel, as to produce in the soap clear-cut and even letters, more especially when the characters are in relief. The workmanship of the die is shown at once in the appearance of the pressed soap. A good die greatly improves the appearance of an inferior product. Toilet soap is stamped on foot-presses, the automatic press having not as yet proved satisfactory for stamping milled soap.

30. The Wrapping of Soap.—The chief care involved in the final process of soap manufacture is neatness. A soap wrapper of average skill will wrap, pack, and nail 40 boxes, of 100 bars each, per day. An experienced wrapper can finish upwards of 50 boxes, while a very rapid worker can often exceed 60 boxes per day.

FIG. 10

31. In Fig. 16 is a summary sketch of the essential mechanical appliances of a soap factory. In studying this illustration, the successive processes of soap manufacture may be traced from the lye tank and the stock tank, through the kettle to the crutcher, thence to the frame, to the slabber and the cutting table, and finally through the drying room to the soap press.

SEMI-BOILED SOAPS

32. General Remarks.—The term *semi-boiled*, as applied to soap, refers to a soap that has not been grained, the saponification being completed in one change and the soap strengthened and settled in a following one. As the soap is not grained, whereby glycerine is separated, the kettle will contain, on the completion of the process, all the material that has been added to it. In semi-boiled soap, rosin may be used as an ingredient, but the process is generally used only for straight glyceride stock.

As no impurities are removed in the waste lye formed by graining, it is necessary, in order to produce a superior quality of goods, that the stock used be of good grade. This process is often resorted to for the manufacture of the base for cheap toilet soaps. It is used generally for preparing the soap base for soap or washing powders. This process is more quickly carried out than is the manufacture of settled soap. The stock can be killed (saponified) in the early forenoon, strengthened after dinner, and pumped to the crutcher, if a filled soap or a soap powder is to be made, or directly into the frame, if intended for toilet soap, on the following morning. If it is desired to save the glycerine and to remove the impurities as well, the soap may be grained sharply after the stock has been killed, the waste lye withdrawn on the following morning, and the soap carefully strengthened, settled, and pumped from the kettle on the third day.

33. The semi-boiled process is primarily a cheap method of soap manufacture, with economy in fuel, labor, and time.

It is best, however, to give the soap a simple purification by graining it sharply, thus prolonging the time consumed in manufacture by one day. By doing so the volume and depth of color of the niger is very considerably reduced. The semi-boiled process is used as well for the manufacture of soft soaps, of which textile soap is a variety, and for green Castile soap made from olive-oil foots. A desired characteristic of the latter soap is the green color arising from the green coloring matter, or chlorophyl, present in the rind and pulp of the fresh olive. This soap is never grained. In graining, the coloring matter would be discharged into the waste lye, with the result that the finished soap would bleach rapidly and unequally on exposure, thus deteriorating in appearance.

SOFT SOAP

34. The Manufacture of Soft Soap.—As the semi-boiled process is used exclusively for the manufacture of soft soap, we will discuss the boiling of this quality of soap in outlining the practical features of the process.

Straight tallow or good grease stock, to which may be added some cottonseed oil, if the price of the latter will permit, is run into the kettle on open steam. For saponifying 10,000 pounds of tallow with caustic-soda lye of a density of 20° Baumé, made from 74-per-cent. caustic, we learn by reference to Table XIII, *Manufacture of Soap*, Part 1, that 97.95 pounds will be required. As tallow is a stock that is easily killed and gives well-defined indications of conditions in the kettle, practically all of this lye may be added by the time the stock is in, saponification, however, progressing in the meantime. The chief care is to avoid bunching by vigorously boiling the contents of the kettle. Open steam is used throughout the process.

In the manufacture of settled rosin soap, we produced a hard soap; here we wish to turn out a soft soap made with a soda base. According to the old definition, a soft soap is one in which the alkali used is potash. That definition is

obsolete, for the hardness of a soap does not depend primarily on the nature of the alkali present, but on the degree of hydration of the soap. A hard soap can be made of potash and olein, which were the ingredients of the original "Castile" soap.

With settled hard soap, a yield of 50 per cent. is generally allowed; with soft soap, a yield may be obtained varying from 225 to 240 per cent. In fact, the yield may be any amount up to 400 per cent., according to the purpose for which the soap is intended.

35. After the stock has been thoroughly killed, open the soap slightly on caustic (the meaning of *opening* is explained in *Manufacture of Soap*, Part 1) and boil. This treatment is equivalent to a strengthening change without the intermediate graining. After having boiled quietly on this strength for some time, with an occasional addition of caustic lye as it has been absorbed, add water gradually until the soap closes. We now have in the kettle the soap-maker's 50-per-cent. yield, and the soap is in a condition similar to that of settled soap on the settling change. The degree of hydration is determined either by the specifications of the purchaser or by the price obtained for the product. After the soap has been closed water is added slowly, care being taken to boil it thoroughly through the mass after each addition, until the soap has been brought to that consistency determined by experience.

A more satisfactory method, and one depending less on the variable and confusing conditions in the soap kettle, is to add the regulated amount of water to the soap in the crutcher. After thoroughly incorporating the addition in the crutcher, the soap is dropped into tight, weighed barrels and allowed to cool. The barrels are then headed and weighed. There is no market in this country for package-made soft soap. It is sold in bulk to institutions for cleansing purposes and in greater quantities to textile manufacturers.

36. When the soap is hydrated in the kettle, the yield is variable, owing to the difficulty of determining the loss of

water by evaporation, and in successive boils may vary several per cent. In the manufacture of soft soap with unmixed stock, as tallow alone, there is not sufficient difference in the amounts of the various glycerides present to produce the *figging* so often desired in a soap of this character. With soft soap made from cottonseed oil, with a small proportion of tallow, the *figged* appearance on cooling may easily be obtained in virtue of the different solidifying points of soaps made from glycerides of different melting points.

Soft soap made from caustic potash will admit of a higher yield within the limits of the quality of the product desired. This arises from the greater combining weight of caustic potash, the molecular weight of caustic potash being 56, while that of caustic soda is 40; also from the fact that a potash soap in a hydrated condition will stand a greater amount of saline filling than will a soda soft soap.

Rosin is used to a large extent in soft soaps, but it should never be present in textile soaps. Linseed oil is largely used in European practice, and, in fact, may be said to be the chief ingredient. Soft soaps admit of sophistication to a high degree, the principal cheapeners and adulterants being aqueous solutions of sodium silicate, soda ash, and potassium chloride; starch is also largely used.

37. Calculation of Yield.—To ascertain the net percentage of yield, it is simply required to divide the net weight of soap made by the weight of soap stock used. To arrive at the total yield, the numerator is increased by the weight of filling material added to the soap in the crutcher or in the kettle.

COLD-PROCESS SOAP

38. General Remarks.—In the discussion of the settled and semi-boiled processes of soap manufacture, it was learned that the latter is the more economical process, and for the manufacture of soaps of a certain character, it is the preferable process. The ease with which the soap can be purified

by graining and removing the waste lye containing the impurities, indicates how loose is the line of demarcation between the two processes. In the discussion of the manufacture of soap by the cold process, we are introduced to a method that surpasses all others in the economy of every element entering into the cost of production. The method, however, possesses certain paramount disadvantages, which restrict its use to a very limited field, notwithstanding the large amount of soap of this character that is produced. The mechanical equipment required consists simply of tanks containing the fat, oil, and caustic-soda lye, a crutcher in which the ingredients are mixed, and frames to receive the mixture and in which the chemical reaction of saponification continues, if under favorable conditions, to completion.

It will be evident that with such simple and comparatively inexpensive factory equipment, that the proportion of fixed charges in the cost of the product is very small. Experience is the chief and most important asset, and without it, notwithstanding the simplicity of the process, satisfactory results cannot be assured.

39. The term **cold process** is a loose descriptive. The saponification is not conducted in the cold, for the heat generated by chemical combination is considerable. The term has reference chiefly to the fact that the instrumentality of heat is not employed, as in the two general processes previously described.

The theory of the process is very simple and essentially demands intimacy of mixture of the ingredients taking part in the reaction. To effect this, the fat or oil must be in a liquid state and the caustic-alkali solutions maintained at such a temperature that when the stock and alkali are mixed, neither will cool the other to stiffness before every particle of the glyceride is brought into contact with the alkali. Even in the daily working of the process, it is a very difficult matter to maintain uniform conditions and to insure the complete absorption of the alkali. As the process is ordinarily practiced, saponification is invariably incomplete,

with more or less free fat and free alkali remaining in the finished product. This is the chief disadvantage of the process, and is the one which greatly restricts the use of cold-process soap for toilet purposes.

40. To secure satisfactory results in the practical working of the process, it is necessary that the caustic alkali be of high grade, not of lower quality than 76 per cent., and that the glyceride stock be fresh and pure. Without these primary qualifications good results under no circumstances can be assured, and with them the prime essentials of satisfactory work are obtained.

Coconut oil, either alone or in admixture with tallow and cottonseed oil, is chiefly used for cold-process soap. The rapid absorption of alkali at a comparatively low temperature, with the production of a smooth, clear soap that will neither crack nor warp on aging and that will admit the incorporation of a large amount of filling, especially adapt it as a raw material for soap of this character. With inferior grades of coconut oil, boiling upon strong brine with open steam and settling to remove impurities are necessary. The same preliminary purification is also required for tallow.

The manufacture of cold-process soap received a great impetus from the manufacture of high-grade caustic soda; the impurities always present in low-grade caustic interfered greatly with the satisfactory working of the process.

THE MANUFACTURE OF COLD-PROCESS SOAP

41. To explain the process in practical detail, we will discuss the manufacture of cold-process soap from 500 pounds of coconut oil. By referring to Table XII, *Manufacture of Soap*, Part 1, we learn that coconut oil will absorb 17.6 per cent. of chemically pure caustic soda. This amount is equivalent to 18 per cent. of 76-per-cent. commercial caustic; therefore, 90 pounds of caustic soda of this grade will be required for saponification.

A caustic-soda lye of 35° Baumé, made from 76-per-cent. caustic, contains 28.28 per cent. of sodium hydrate.

Therefore, to furnish 90 pounds of sodium hydrate, 318 pounds of 35° Baumé caustic lye, made from 76-per-cent. caustic, will be required.

The purified coconut oil at a temperature of 115° to 120° F. is run into the crutcher and the latter put into agitation. The required amount of lye is then run in and crutching continued until a portion of the mass, removed on a paddle, shows a clear, homogeneous appearance. This condition can be ascertained only by experience, and under no consideration should the contents be removed from the crutcher until the most thorough mixing possible has been effected. The mixing period need not exceed 20 to 30 minutes, and with experienced working the time required is usually less. The mass is then dropped into frames, which are removed and carefully covered with soda-ash bags or other suitable material to prevent too rapid cooling. The frames should not be exposed to currents of cold air nor in any way subject to rapid cooling.

42. There were added in the crutcher 500 pounds of coconut oil and 90 pounds of solid caustic dissolved in 228 pounds of water. This mixture yields a soap which, in so far as hydration is concerned, corresponds to the composition of unfilled settled soap. We have obtained a yield of practically 63 per cent. By adding tallow and cottonseed oil in suitable proportions, the cost of the soap may be greatly reduced. The yield may be increased and the cost in this manner again greatly reduced by the addition of sodium silicate, soda ash, pearl ash, mineral soap stock, starch, or talc.

All the filling material in cold-process soaps is generally mixed with the lye previous to being added in the crutcher. If it is desired to perfume the soap, the essential oil is added directly to the crutcher towards the end of the mixing process.

It should be borne in mind that the more highly filled a cold-process soap is, the more is the process of saponification retarded through the presence of inert material. Cold-process soap will admit of more filling than will settled

soap without a corresponding deterioration of appearance on aging. This exceptionally high absorption of saline material by coconut oil is explained on the same basis as the large amount of salt required for graining coconut-oil soap made by the settled process.

The substitution of caustic potash for a portion of the caustic soda is desirable, as a clearer, smoother, and milder soap will result.

43. In the procedure described above there has been no interference in the process on the part of the attendant. The ingredients have been mixed in the calculated proportions and the mixture so obtained treated mechanically in the hope that the results will be satisfactory. As experience in the operation of the process is acquired, the treatment may be modified according to the judgment of the attendant. In the following procedure the judgment of the manufacturer is called into play.

Add the coconut oil at a temperature of 115° F. to the crutcher and start it slowly and then add gradually the caustic-soda lye, which has been prepared at the same temperature. When all the caustic-soda lye has been added, continue crutching at the same or only slightly greater speed for a period of 5 minutes. At the expiration of this period, stop the crutcher and enclose it with a cover and soda-ash bags to retain the heat. Allow the mass to stand for an hour and then start the crutcher slowly. If a simple mixer of style A, Fig. 11, *Manufacture of Soap*, Part 1, is used, the contents should not rise above the level of the topmost horizontal arm. If they do, this portion of the contents will not be thoroughly mixed. With a crutcher of style B, Fig. 12, *Manufacture of Soap*, Part 1, the contents should not rise above the level of the central cylinder enclosing the screw. With a steam-jacketed crutcher, the process can be more easily controlled through the use of steam, should the temperature fall below that ascertained by experience to be best, viz., 160° F. If at this stage the soap should show an excessive sharpness to the tongue, a

few pounds of coconut oil may be added and crutched in until the soap tastes free from caustic. On the contrary, should the soap, after the maximum heat of the reaction has been evolved, be neutral to the taste, a few pounds of 15° Baumé caustic-soda lye may be added and thoroughly incorporated. After these adverse symptoms have been corrected, start the crutcher at full speed and continue crutching for fully 20 minutes, until the mass rises white and smooth to the top of the crutcher. The perfume is now crutched in and the soap framed.

44. Manufacture of Cold-Process Soap Directly in the Soap Frame.—It is possible to make a fairly satisfactory cold-process soap directly in the soap frame, thereby eliminating the crutcher. Elkington has given instructions for the manufacture of cold-process soap by this method according to the following formulæ:

TABLE II

FORMULAS USED IN MAKING COLD-PROCESS SOAP

Ingredients	Formula No. 1. Pounds	Formula No. 2. Pounds
Tallow.....	75	75
Coconut oil (Ceylon).....	25	25
Caustic-soda lye (35½° Baumé) made from 74% caustic.....	75	70
Silicate of soda, N grade.....	125	100
Pearl-ash lye, 36° Baumé.....	20	17
Amount of soap produced....	320	287

Weigh out the proportions of tallow and coconut oil required for a frame of soap into a tight frame. Weigh out the quantity of caustic-soda lye required into a separate vessel, also weigh out the proportion of silicate of soda needed into another vessel. The pearl-ash (crude potassium carbonate) solution is then weighed out and mixed with the

silicate of soda. When all is ready for mixing, the temperature of the tallow and coconut oil in the frame should be 145° to 150° F. in cold weather and 125° to 130° F. in warm weather. The caustic-soda lye and silicate-of-soda mixture are at the normal temperature of the factory.

When the temperature conditions are as stated above, the caustic-soda lye is run alone and quickly into the frame containing the mixed tallow and coconut oil, the mass in the meantime being crutched vigorously from the bottom of the frame. After adding the caustic-soda lye, crutching should be continued until the mass begins to thicken. The mixture of sodium silicate and pearl-ash lye is now added quickly with continued crutching.

After the last addition, the mass in the frame will be thinned out considerably and with continued crutching will gradually become of a thick, creamy consistency. The two hand crutches are now removed and the frame carefully covered and allowed to stand without disturbance until the soap is cold. If the soap is to be perfumed, the essential oil is stirred in with the silicate-of-soda mixture and added with it. If the frame must be moved from where the soap is made, move it quickly before the silicate-of-soda mixture is added; then add it at once and finish the soap as directed.

The cold process is primarily a quick process, the tardy addition of any ingredient being sufficient to mar the results.

For a 1,000-pound frame, the caustic-soda lye must be run into the soap stock in from 90 to 120 seconds. The addition of the silicate-of-soda mixture should also not require more time than this. Two crutches (see Fig. 3) should be used and the work carried on quickly and the bottom of the frame reached at each stroke. With satisfactory conditions of temperature, the time required in preparing a frame of cold-process soap, as outlined above, should not exceed from 12 to 16 minutes. Care must be taken not to crutch the mass too long. To insure a smooth soap, crutching should be stopped as soon as a mark made on the surface of the soap will remain, and under no circumstances should the frame be disturbed until the contents are cold. With good, firm

tallow, cottonseed oil may be substituted for it to the extent of 30 to 50 per cent., and less if the tallow is softer. With soft stock, longer time will be required for cooling.

45. The apparatus used in cold-soap manufacture admits of very convenient arrangement. The soap stocks and alkali tanks should be placed on an elevation above the crutcher and should be provided with closed steam coils for heating the contents to the required temperatures.

The soap stock and alkali can then flow by gravity into the crutcher. The sides of these tanks should be graduated into vertical inches and the cubical capacity of the tank corresponding with them, as well as the weight of the material contained therein, should be plainly indicated. By this means, mistakes in the weights of the material used may to a great extent be avoided.

46. In summing up the advantages and disadvantages of the cold process, it may be stated that the soap is simply, easily, and quickly made, with but little outlay required for plant and labor. It admits of a greater yield than that furnished by any other process for the manufacture of hard soap. A well-made cold-process soap has not only a better appearance than a boiled soap, but retains its good appearance longer. It admits of a greater degree of sophistication than does a settled soap. The process differs greatly from the boiled processes in one important particular, viz., that small quantities can be made at a time. The process is used largely for cheap toilet soaps, in some instances for laundry soap, and almost entirely in the manufacture of chipped soap for sale to laundries.

As ordinarily practiced, the process does not admit of complete saponification, as does the boiled processes, the soap invariably containing, according to the care and experience used in its manufacture, greater or less amounts of free caustic alkali and uncombined fat or oil. Rancidity soon develops from the presence of uncombined oil. This is the chief, and practically the only, disadvantage of the cold process.

REMELTING SOAP

PURPOSE OF REMELTING

47. General Remarks.—Soap scraps result from various sources, which may be enumerated in the order of their occurrence, viz., frames of soap that have cracked or fissured or for any cause are not suitable, either in whole or part, for slabbing and cutting; scrapings from frame bottoms; defective bars and trimmings from the cutting table; spoiled bars from the soap press; and unsatisfactory boxed soap returned by the trade. This soap contains its proportion of the filling added in the crutcher and which, if the soap were returned directly to the soap kettle, would be lost in the waste lye. The function of the remelter is to remelt these scraps, and when melted the soap is crutched and framed as before.

Some perfume and usually a small quantity of soda-ash solution are usually added to the remelted soap in the crutcher. The texture of remelted soap when cut and pressed differs from that of freshly pressed soap in being less clear.

48. Remelters.—The remelter shown in Fig. 17 consists of a sheet-steel tank with a system of closed steam pipes *b* arranged vertically inside and also a system of closed steam pipes *c* connected with *b* arranged horizontally on the bottom and a short distance above the outlet *e*. A coarse wire netting *d* serves as a strainer. It is placed below the steam coils *c* and above the open steam jet *f*. The pipe *i* carries steam to the upright system *b*; the exhaust pipe *j* carries the water of condensation from the horizontal system *c*; the live-steam pipe *k* supplies the steam-jet *f*. The remelter is usually covered with insulating material to retain the heat and is surmounted by a wooden casing to receive the soap on the floor above. The steam pipes are so arranged as to distribute the heat uniformly

throughout the mass and to interfere least with the flow of the melted soap, by gravity, from the bottom of the remelter.

49. When sufficient scrap for several frames has accumulated in the remelter, steam is admitted into the closed coils and the adjacent soap melted. Live steam is then introduced for a period of 10 minutes in addition to the closed

FIG. 17

steam. At the expiration of this period, remelted soap should flow freely from the outlet. The live steam is now turned off. When sufficient of the mass has melted to charge the crutcher, which is usually placed below the remelter in factories where a separate remelter is used, framing is begun and continued as fast as the soap melts. The remelted soap withdrawn from below is

replaced by unmelted soap, which sinks by gravity from the wooden casing surmounting the remelting tank. The remelter is an indispensable part of the factory equipment.

When the size of the factory does not warrant the installation of a separate remelter, as shown in Fig. 17, it may be combined with the crutcher, as shown in the remelting crutcher in Fig. 18. The remelting crutcher, Fig. 18, is

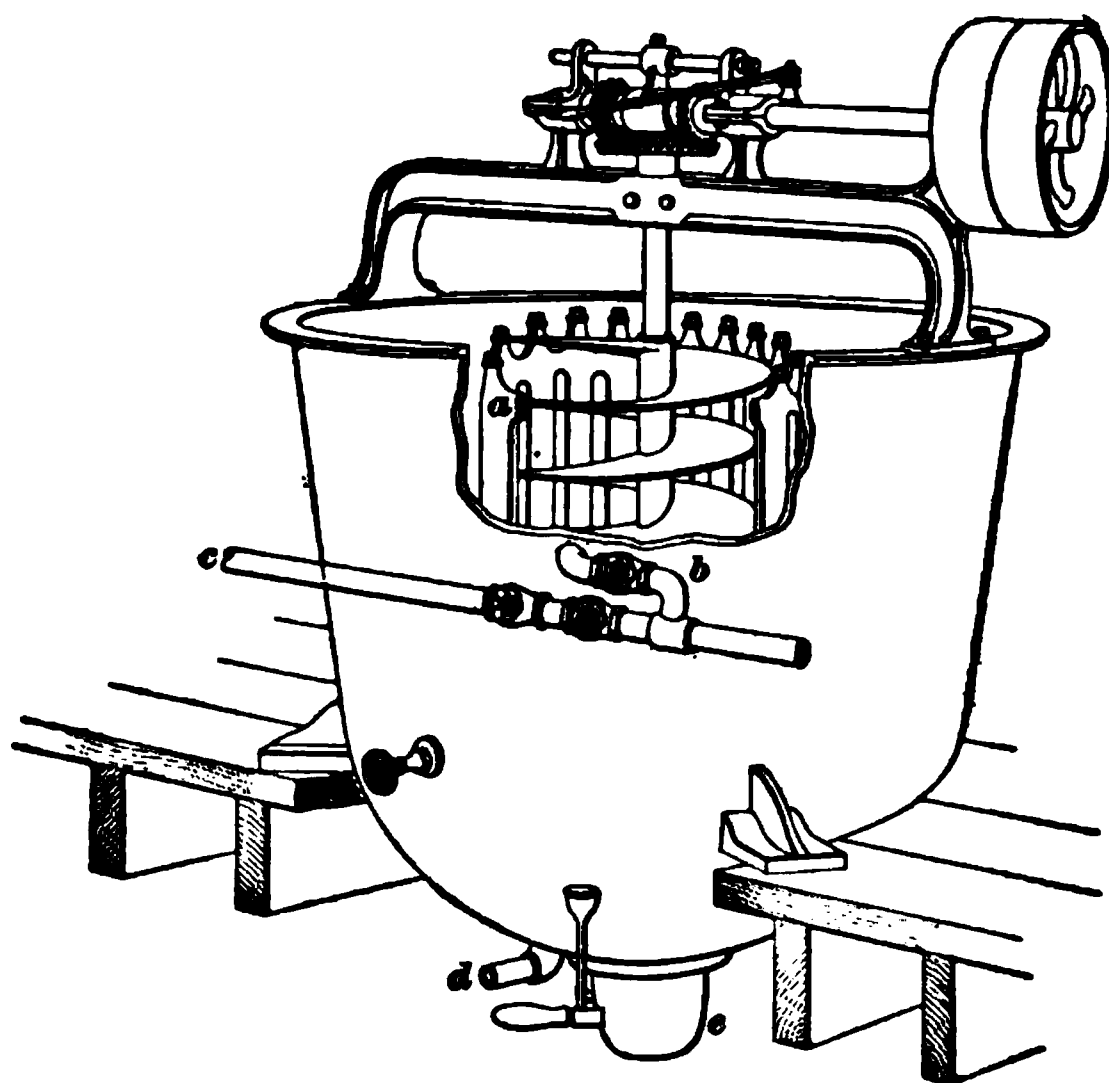


FIG. 18

simply a crutcher of style B, shown in Fig. 12, Part 1, in which the inner concentric cylinder is replaced with vertical closed steam pipes surrounding the screw. Steam is admitted into the vertical closed steam pipes *a* through the valve *b*. Live steam is introduced at *c* and the water of condensation is withdrawn from the closed steam pipes at *d*. The outlet of the crutcher is at *e*. The remelting of soap in this device is carried on practically as with the regular remelter. Soap scrap may be introduced by gravity from a receptacle placed above the crutcher.

THE MANUFACTURE OF TOILET SOAP

MILLED-PROCESS SOAP

50. General Remarks. — The manufacture of toilet soap requires the installation of expensive machinery and a greater degree of skill than is necessary in the manufacture of laundry soap. Not only the cost of plant and the superior skill required in manufacture, but the experience and ability required in marketing the product, confine its manufacture to a comparatively limited number of plants. The manufacture of toilet soap in the United States dates from 1844, at which time a Frenchman, Jules Haul, made soap of this character with very primitive apparatus, in Philadelphia. The French were the original manufacturers of toilet soap, and with their experience and prestige as perfumers, have been able to maintain their reputation for superior products of this kind to the present day. Since the original manufacture of toilet soap in this country, as noted above, American skill and experience have so far advanced, together with the superior mechanical appliances used in its manufacture, that the toilet soap produced in this country is equal to that produced elsewhere in the world.

51. Although toilet soap can be made by any of the three processes described, the term generally has reference to what is called a **milled soap**, meaning by this a soap that after having been partially dehydrated, is kneaded into films of dough-like consistency in a machine called a *mill*. The soap is then compressed into an elongated bar, which is cut into sections and pressed into separate bars.

By this process of manufacture it is possible to combine the finest style and finish with superior quality and durability. Owing to the partial dehydration to which the toilet-soap base has been subjected, its milling and subsequent compression in the plodder, soap manufactured in this manner is the most economical for toilet purposes.

52. Toilet-Soap Base.—The manufacture of toilet soap requires first the manufacture of what is commonly called the **toilet-soap base**. It may either be a well-made, settled soap, a semi-boiled soap, or a cold-process soap. The principal requirements of a good toilet-soap base are that it must be made from fresh material of good quality, must be free from impurities, completely saponified, perfectly neutral, and of a good tough grain. In large establishments, the manufacture of the toilet-soap base is usually intrusted to one man with special skill in the boiling of soap for this purpose. The nature of the stock used is very important.

Good, fresh tallow, with the characteristic shortness of grain of the soap made from it, softened by the addition of suitable proportions of cottonseed oil, and smoothed with a proportion of coconut oil, will make a first-class toilet-soap base. The formulas of the toilet-soap base vary greatly. Castor oil and lard may be used to make the soap more mild and to admit of a finer finish in the pressed bar. In the manufacture of cheap toilet soap—what is known in the trade as five-and-ten-cent goods—not only is material of inferior quality used, but also adulterants well known to the toilet-soap miller, to which is added a fragrant and lasting perfume. Palm oil forms an excellent ingredient of the toilet-soap base, the natural odor of the oil harmonizing well with the perfuming material.

In the boiling of the toilet-soap base, the greatest care lies in completely saponifying the stock. In the use of coconut and cottonseed oils, this is very important, for there are no oils used by the soap maker that are more liable to become rancid, especially when present as unsaponified stock in the finished soap, the rancidity being invariably betrayed by the odor on the hands after using.

53. Boiling the Toilet-Soap Base.—The manufacture of the toilet-soap base does not differ essentially from the manufacture of settled rosin soap. The stock must be pure and fresh, the kettle and all appurtenances scrupulously clean, and completeness of saponification must be insured.

The stock change is effected as described in Part 1, care being taken to thoroughly kill the stock, even though the stock lye may contain an excess of caustic alkali. A slight strength is desirable, as it is believed that the texture of the soap is thereby improved. After graining and settling the waste lye, the latter is run off. The soap is then boiled, or strengthened, with the addition of weak caustic lye, after which it is grained sharply with undiluted caustic lye. This strength lye is withdrawn and held in storage for use in inferior grades of soap. The soap is now boiled up with live steam and water is carefully added until the soap shows a characteristic flat grain and is all but closed. The soap is now allowed to settle, as on the settling change in the manufacture of settled rosin soap. It is believed that the more completely settled a toilet-soap base is, the better adapted it is for milling, from the separation of salts and impurities that tend to form the niger. The more thoroughly these are removed, the less liable is the soap to come cracky from the plodder. At the expiration of the settling period the soap is pumped directly into frames.

54. The nature of the process used in the preparation of the toilet-soap base and the care taken in the operation are determined by the quality of the product desired. For cheap, milled soaps, a base made by the cold process may be used, but more generally the semi-boiled process is used. With the latter process the soap should be carefully settled, and the niger, if stock of good quality is used, can be incorporated in a succeeding boil or preferably added to a kettle charge for laundry soap. The soap is framed in the regular way and when cold is ready for cutting.

In the manufacture of hard soap, free from filling, whether by the settled, semi-boiled, or cold process, the yield is commonly estimated as 50 per cent. This, therefore, gives the toilet-soap base, by whatever process it is manufactured, a water content varying from 30 to 33 per cent. To insure a satisfactory product on milling, this percentage of water must be reduced about 50 per cent. With settled soap for laundry

purposes, the water content was reduced from 3 to 5 per cent., just sufficient to permit a good finish on pressing and stamping.

55. Preparation of the Toilet-Soap Base for Milling.

The frame of soap is cut into long bars of such dimensions

that the preliminary drying is effected in the quickest manner.

At this stage it is merely desired to dry the soap, so that it can be reduced to chips in a cleanly manner.

The superficially dried bars of soap are laid lengthwise in the feed-box *a* of the automatic chipper shown in Fig. 19, whence they fall by gravity against the knives set in the radial slits *b*, two of which are shown. The soap can be cut into chips of any desired thickness by adjust-

FIG. 19

ing the knives at varying distances above the surface of the disk *d*, which is rotated by a pulley on the shaft *c*. The chips are collected on the opposite side of the machine and spread on the bottoms of trays. These are then transferred to the drying room, where they are allowed to remain until the water content has been reduced to that degree permitting the most satisfactory treatment in the milling machine.

A very convenient arrangement for the drying of toilet-soap chips is shown in Fig. 20. Air is drawn through the sectional heater *a* in the direction indicated by the arrows by

means of the centrifugal fan or blower *b* shown in outline by the dotted lines, and forced through the casing of drawers and expelled at the stack. The degree of dehydration is variable, depending on the quality of the product desired.

If cheapening material is to be added in the mill, its capacity for moisture demands that the chips be not too dry, or the soap will come cracky from the plodder. Again,

FIG. 20

with unfilled toilet soap, the consistency of the mass may be varied at will by suitable treatment during the milling process. If the soap works too dry in the mill, either softer soap of the same quality or some water may be added, according to the judgment of the miller.

The continuation of the drying process is best determined by experience. For goods of superior quality, the chips are sufficiently dry when the water content has been reduced from 30 to 33 per cent. down to 15 or 18 per cent. The chips are now ready for the milling process.

56. Toilet-Soap Mill.—The toilet-soap mill shown in Fig. 21 consists of a hopper *a*, in which the soap to be milled is placed, set upon *b* and *c* of a series of granite rolls *b*, *c*, *d*, and *e*. These rolls vary from three to five in number.

They are carried upon heavy steel shafts, forming the core of the roll, and mounted in the most improved machines one above the other at an angle of 45° . Quincy granite, with a smooth and true finish, is generally used for the rolls, which vary in diameter from 8 to 18 inches, and in length from 16 to 24 inches or longer, as desired. The capacity of the mill is rated by the manufacturer according

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FIG. 21

to the amount of soap milled per hour, varying from 80 to 250 pounds for the roll dimensions mentioned above. The roll *c*, Fig. 21, is supported in stationary journals; the intervening distances between adjoining rolls may be varied at will by means of setscrews on both sides of the mill, and shown on one side at *f*, *g*, and *h*. The rolls rotate upon their shafts in the directions indicated by the arrows.

57. Milling the Tollet-Soap Base.—The chips, suitably dried and with the right "feel" as they are taken up in bunch by the hand, are introduced into the hopper to the amount required for a charge and passed through the mill until they have been reduced to a fairly homogeneous mass. One milling should suffice for this initial reduction.

During this preliminary milling, the distance between the rolls should be greater than at later stages in the milling process, owing to the coarseness of the material worked. During milling, the chips pass down between rolls *b* and *c*, Fig. 21, up between rolls *c* and *d*, down between rolls *d* and *e*, and then, in the condition of a thin, translucent film, they are removed by scrapers from the last roll and returned to the hopper, if desired, for another milling.

When of proper consistency, the soap is collected in a box or trough, to which is added the perfume and coloring matter in proportions based upon the weight of the charge of soap delivered to the mill. If the perfume and coloring matter are in a dry state, it may be simply dusted on the soap in the mill. If liquid, they must be added to the shreds of soap resulting from the first milling and allowed to percolate through, yet not flow from the mass. The soap is then transferred to the hopper and worked through the mill to the satisfaction of the attendant.

For cheap goods, three to four millings may suffice; for superior goods, or to produce the best possible texture in goods of any quality, seven to eight millings are required. After the milling process, the shreds of soap should be perfectly homogeneous, with the perfume and coloring matter, if any of the latter is used, thoroughly worked through.

58. Plodding Toilet Soap.—After satisfactory results have been obtained in the milling process, the soap is ready for the plodder. The soap as it comes from the mill is in thin, translucent laminae. The function of the plodder is to compress this soap into a compact mass, which is expelled from the plodder in the form of an elongated bar, which is then cut into cakes of dimensions adapting them to the mold and die of the soap press.

59. Plodder.—The earliest form of plodder was the naked hands, which molded the mass, crudely kneaded with mortar and pestle, into a globular or oblong bar. The earliest form of machine plodder was a cylinder enclosing a

screw, by means of which the soap, fed into the cylinder at one end and above the screw, was compressed.

This is essentially the construction of the modern plodder. The improvements upon this early form consist chiefly in an increase and a more scientific distribution of the weight of the machine, and in the substitution of steam as the source of power, thus permitting a greater compressive force to be applied to the soap. These mechanical improvements in the plodder have enabled the toilet-soap manufacturers to produce a more compact, a more durable, and a more handsome piece of soap.

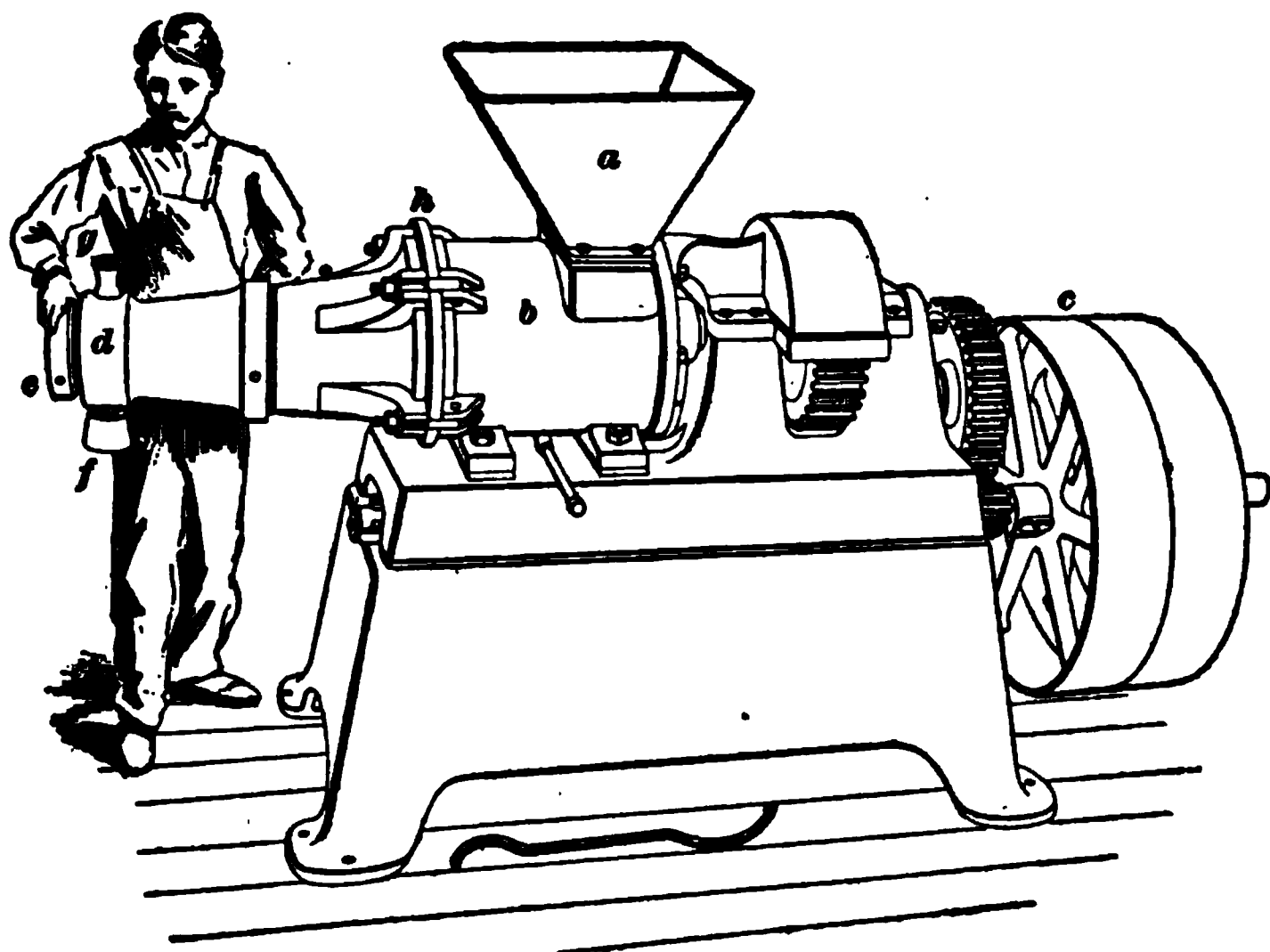


FIG. 22

In the plodder shown in Fig. 22, *a* is the hopper in which the soap is placed, *b* is the case enclosing the horizontal screw compressor impelled by power applied by belt to the pulley *c*, *d* is a concentric hollow casing enveloping the nozzle of the plodder, and is filled with water added at *g* and maintained at a definite temperature. The cake former *e* is heated by a gas flame applied at *f*; *e* is screwed to the nozzle through which the elongated bar of compressed soap

is forced. A sectional connection *h* of the screw case provides for the ready examination of the interior of the case. So long as soap is added to the hopper, the action of the plodder is continuous.

Plodders may be divided into two classes, according to the mode of applying the power—viz., the *hydraulic* and the *steam plodder*. With the former, the output per day does not exceed 1,000 to 1,200 pounds of soap; with the latter, the output in the same time is much greater. The hydraulic plodder is being displaced by the steam-driven plodder, with its greater rapidity of operation.

FIG. 23

The nozzle of the plodder is provided with a screw head, to permit the attachment of the cake former, whereby the elongated bar is made, to assume any cross-section desired, as circular, square, or rectangular. The purpose of the hot-water jacket, shown at *d*, Fig. 22, is to soften the soap to that consistency that, when forced through the cake former, the cohesion of the soap mass will not be destroyed and the

soap, as it is termed, "crack" or "splinter." Plodders are rated according to the pounds of soap capable of being plodded per day under a maximum average compressive force.

60. Cutting and Pressing the Plodded Toilet Soap.

The soap as it is delivered from the nozzle of the plodder is carried to the cutting table, shown in Fig. 23, where the long bar is cut into separate cakes of uniform length by the wire *a* in the movable frame *b* operated by foot-power. The experience of the attendant will determine at this stage if it is necessary to subject the soap to a slight superficial drying in order to obtain the best possible impression on stamping. If the soap is sufficiently dry, it is received from the cutting table in trays and carried to the soap press. Milled soap has always been pressed on the old-style foot-press, but with the improved automatic press, it is claimed that soap of this character can be satisfactorily pressed. From the press, the bars are delivered to girls, who wrap and pack them in boxes.

THE PERFUMING OF SOAP

61. General Remarks.—Severe competition in the toilet-soap business has reduced the profit to a very small margin. One important characteristic of the toilet-soap business is the part played by the manner in which the goods are put upon the market. An attractive wrapper and an agreeable perfume are primarily demanded by the inexperienced buyer of toilet soap to the entire exclusion of the essential qualities. By means of coloring matter and suitably blended essential oils, a milled soap, made from very inferior stock, may have its original inferiority completely obscured.

The blending of essential oils is a recognized art, whose development requires the constant attention and study of the skilled perfumer. The demand is always for something new, to supply which the toilet-soap manufacturer is often

hard pressed. Organic chemistry has supplied the perfumer with a large and increasing number of artificial perfumes, which often surpass the natural source in intensity of fragrance and cheapness. Some of these, as nitrobenzene or oil of mirbane, a cheap and efficient substitute for the oil of bitter almonds for perfuming laundry soap, are obtained from coal tar; others, as vanillin, borneol, menthol, eugenol, ionone, heliotropin, etc., are prepared synthetically; certain others are isolated from essential oils, as safrol, from oil of camphor, which has practically displaced the natural oil of sassafras as a soap perfume; citral from oil of lemon; carvol from oil of caraway, etc.

PERFUMES USED IN LAUNDRY SOAPS

62. The following list of essential oils comprises those most commonly used for perfuming laundry soap. As has been noted, the proportion used seldom exceeds $2\frac{1}{2}$ pounds per frame of 1,200 pounds of soap.

Oil of caraway seed.

Oil of cassia, with guaranteed percentage of cinnamic aldehyde.

Oil of cedarwood.

Oil of citronella.

Oil of mirbane, artificial, with odor of oil of bitter almonds.

Oil of rosemary.

Safrol, artificial oil of sassafras.

Oil of eucalyptus.

Oil of thyme.

The best way to ascertain the most satisfactory combination of essential oils for a laundry-soap perfume is to prepare a number of mixtures, to be experimentally used as a basis or vehicle for a highly fragrant and strong-bodied essential oil, as artificial oil of sassafras, or safrol. This particular oil commends itself because of its cheapness. Its fragrance may be modified by mixing it in varying proportions with other essential oils not debarred by their high cost, until a

mixture is obtained that is at once cheap, lasting in fragrance, and disguises most completely and agreeably the odor associated with the soap in use.

After two or three mixtures of equal satisfaction are obtained experimentally, the mixtures may be subjected to practical tests by studying results, when they are mixed in regulated proportions in successive frames of soap.

The following formulas represent two mixtures, No. 2 being modified from No. 1 as the result of an advance in the price of cassia oil. Equally satisfactory results were obtained from both mixtures.

TABLE III

FORMULAS FOR PERFUMING LAUNDRY SOAP

Essential Oils	Formula 1. Parts by Weights	Formula 2. Parts by Weights
Safrol.....	23	27
Cedarwood.....	12	10
Cassia.....	9	7
Lavender.....	10	10

Laundry soap is seldom if ever artificially colored. Unbleached palm oil is the cheapest and most efficient agent available to disguise the odor and color of rosin. Very small quantities of ultramarine are used in some cases to heighten the whiteness of floating soaps. Disinfectant (so called) and tar soaps are colored, the former variably, the latter with pine tar.

THE PERFUMING OF TOILET SOAP

63. The direct application of the perfuming material to the mass of thin, shredded soap after its preliminary treatment in the mill, to obtain homogeneity, involves no especial

difficulty, it being merely sufficient to add the weighed or measured quantity of perfume to the mass in the box, in which it accumulates as received from the mill, and to incorporate it sufficiently with the soap to enable the attendant to transfer it to the hopper of the mill without waste.

The blending of perfuming material to obtain desired odors, generally those imitating the natural odors of fresh flowers, constitutes a distinct art and requires for proficiency an intimate knowledge of the chemistry of essential oils and experience in working with them. The various ingredients of the perfume stock may be bought separately and compounded or blended by the perfumer, or they may be bought already blended from dealers in essential oils and perfumers' materials.

With the exception of musk and civet, which are excretory secretions of the animals producing them, the raw materials of soap perfumery consist chiefly of essential oils obtained from wood, as oil of cedarwood; from the bark, as the oil of wild cherry; from the foliage, as oil of eucalyptus; from the fruit or seed, as oil of caraway; and from the flowers, the chief source, as oil of rosemary.

There is a large and increasing number of synthetic preparations and isolated bodies possessing in a marked degree the fragrance of the natural source that now successfully compete with the latter. These bodies are, as a rule, the odoriferous principle of the natural oil, and for purposes of perfumery are not encumbered with the non-essential ingredients present in the natural oil. A correspondingly smaller quantity can be used to effect the same results and generally on the side of economy.

64. The fractional distillation of essential oils obtained from plants has shown that they are composed of a nearly odorless vehicle, consisting of one or more hydrocarbons of the terpene class, a principal odorous constituent, which may be an alcohol, phenol, aldehyde, ketone, ether, or ester, and

smaller quantities of various other compounds. These modifying constituents may vary in amount in oil obtained from different parts of the same plant.

In Table IV the essential oils are classified according to their chemical composition and principal constituents.

TABLE IV

CLASSIFICATION OF ESSENTIAL OILS

Families	Principal Constituents	Oils
I. Alcoholic Terpenes and their Ethers	1. Borneol 2. Linalool: dextrogyrate levogyrate 3. Geranoil and citronel- lol 4. Menthol 5. Sesquiterpenic alcohol	Pine needle Valerian Linalo6 Coriander Bergamot Lavender Aspic Sage muscatelle Neroli Petit grain Limetta Ylang-Ylang Cananga Palma rosa Geranium Rose Citronella Peppermint Patchouli Sandal wood Angostura bark Caparrapi
II. Aldehydes	1. Benzoic aldehyde 2. Cumic aldehyde 3. Cinnamic aldehyde 4. Citral and citronella	Bitter almond Wild cherry Cherry laurel Cumin Eucalyptus hæmastoma China cinnamon, cassia Ceylon cinnamon, cassia Lemon grass Melissa Citron Petit grain (lemon) Orange Mandarin Lemon Euca-lyptus { Backousia citrio- dora Staigeriana Dealbata Maculata

TABLE IV—(Continued)

Families	Principal Constituents	Oils
III. Ketones	1. Methylnonylketone	Rue
	2. Irone	Iris
	3. Carvone	Caraway Dill Kuro-moji Spearmint Mint crépue
	4. Putegone	Pennyroyal Pennyroyal (American) Thyme
	5. Thuyone	Tansy Thuja Wormwood Sage Artemesia (barrelieri)
	6. Fenone	Fennel
	7. Camphor	Camphor laurel Rosemary Marjoram
IV. Lactones and Anhydrides	1. Alantolactone	Elecampane
	2. Sedanolid and sedonic anhydride	Celery
V. Phenols and Phenolic Derivatives	1. Thymol and carvacrol	Ajowan Thyme Wild thyme Monarde Satureja Origanum Curcuma
	2. Eugenol and betel-phenol	Clove Massoy bark Canella bark Bay Gulilivan Allspice Bétel Para-coto bark Snake root (European) Snake root (Canadian) Matica
	3. Anisol and estragol	Anise Anise bark Estragon Persea Basilicum
	4. Safrol	Sassafras Oil of camphor
	5. Apiol	Parsley Camphorwood (Venezuela)
VI. Phenolic Aldehyde	1. Salicylic aldehyde	Reine des Prés
	2. Diosphenol	Buchu Diosma Creneta

TABLE IV—(Continued)

Families	Principal Constituents	Oils
VII. Cineol		Eucalyptus Cajeput Cardamon Cheken leaf Galongal Laurel Laurel (California) Myrtle Zedoary
VIII. Terpenes and Sesquiterpenes		Turpentine Cypress Angelica Phellandrene Wormseed Ginger Frankincense Elemi Black pepper Cubut Copaiba Gurjun balsam Juniperberry Cedar Hop Hemp
IX. Ethers of Alcohols of the Aliphatic Series		Chamomile Heracleum Parsnip Wintergreen Cognac
X. Sulphur Compounds	1. Sulphides 2. Isosulphocyanates	Garlic Assafoetida Onion White mustard Black mustard Mignonette Cochleria Achillea
XI. Constituents Unknown		Musk pear Mugwort Arnica Carrot Carlina Calamus Cascarilla Erigeron Galbanum Guajacum wood Hyssop Masterwort Jaborandi Jasmine Champaca Lovage Myrrh Tea Veti vert

THE MANUFACTURE OF PERFUMES

65. The essential oil constituting the natural odor of vegetation, whether it be of the wood, bark, foliage, flower, or fruit, may be extracted by any one of five distinct methods—viz., *distillation*, *masceration*, *enfleurage*, *extraction with volatile solvents*, *mechanical means*.

Distillation consists in subjecting the raw material, mixed with water, in a boiler to heat, preferably steam, whereby the essential oil is driven off with the steam that is condensed, allowing the essential oil and water in separate layers to be collected in a receiver. As a process of extraction, distillation involves the greatest danger of decomposition and loss of the product desired. It is usually employed for the extraction of oils from the woody portions of the plant, although the delicate perfumes of the rose and the orange flower will withstand distillation.

Masceration consists in immersing the flowers in melted and very pure lard or tallow. For preparing perfumed oils, olive oil is generally used. The absorption of the odorous principle is hastened by renewing the charge at frequent intervals as absorption progresses. The mass may be used either directly as a pomade or extracted from the fat with alcohol, forming the “extracts” of the perfume trade. Masceration is applied to the violet, jonquil, rose, and orange flowers. The process is usually carried out by women, who keep the flowers submerged in the melted fat, at a temperature of about 65° C., for about $\frac{1}{2}$ hour, by means of spatulas. When sufficiently exhausted, they are removed, drained, and pressed free from adhering fat. The process is repeated with the same fat until it has been saturated to the desired strength.

Enfleurage differs from masceration in that the absorption takes place in the cold. This process is used for the most delicate and elusive odors, as the jasmine and the tuberose. In this process the flowers or petals are laid on thin layers of grease spread out on the glass bottoms of shallow rectangular frames. The separate frames charged in this

manner are then arranged one above another in piles. The flowers remain in contact with the grease for about 24 hours and are then removed and fresh flowers added, which process is continued until the grease is sufficiently saturated. In some instances, as with the jasmine, upwards of 2 to 3 months are required. To make perfumed oils by this process, a cloth

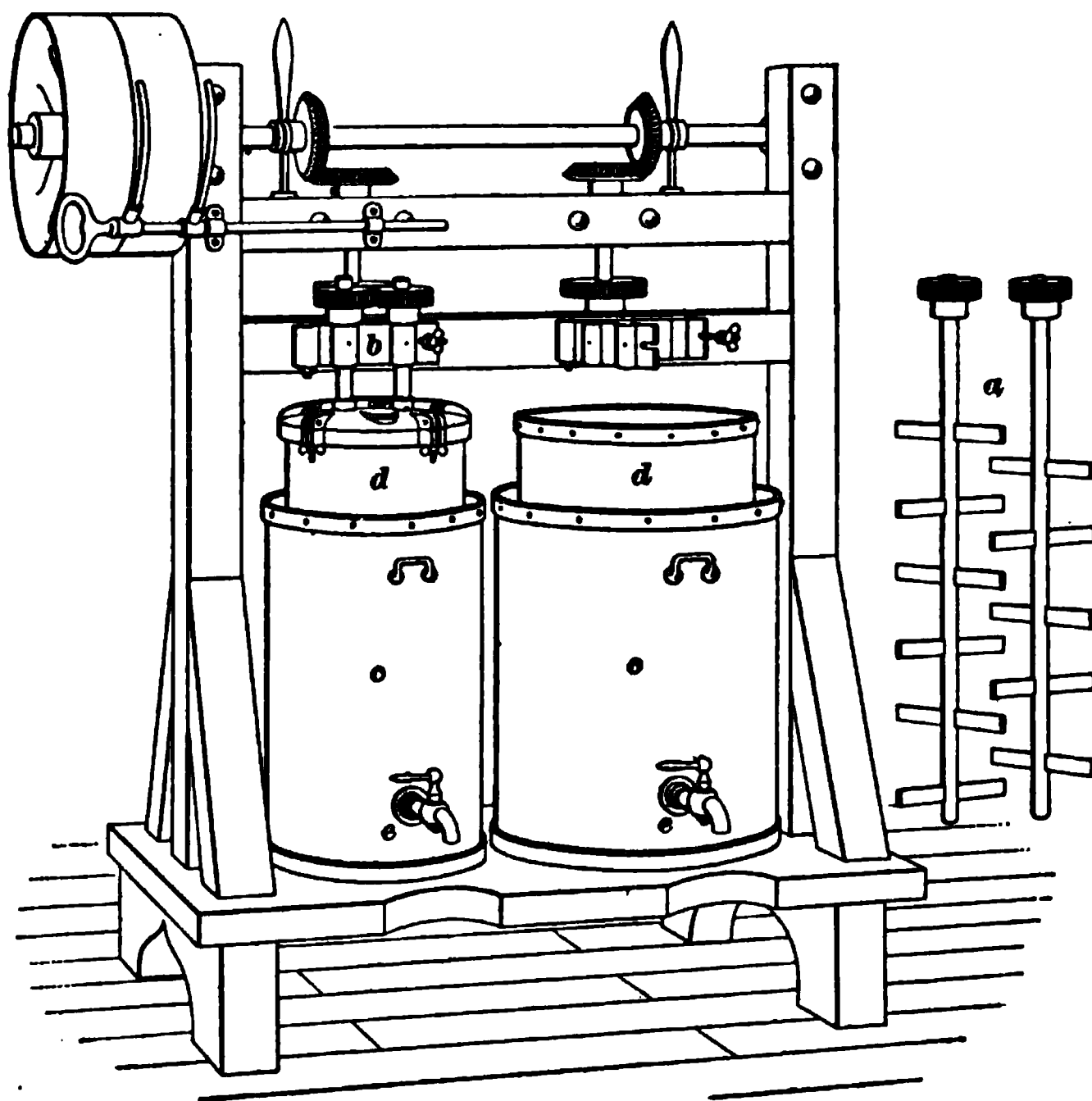


FIG. 24

saturated with olive oil is spread upon the wire-gauge bottom of a wooden frame and receives the fresh flowers. Enfleurage is the most delicate of the five methods and gives the best results.

Extraction with volatile solvents finds very limited application as a process applied directly to the raw material. It constitutes the final treatment of the perfume obtained by

masceration and enfleurage. The scented fat, if solid, is called a "pomade;" if liquid, an "huile antique." The odorous material is separated from the fat vehicle by means of a pomade washer, shown in Fig. 24. This machine is simply a crutcher with detachable mixers held firmly in place by the lock *b*, the whole being enclosed by a water-cooling jacket *c*. The pomade, with the solvent, usually alcohol, is added to the tank *d*, the crutches *a* inserted and firmly locked at *b*. The cover of the crutcher is now firmly clamped down and agitation begun. When solution is complete, the liquid is withdrawn at *e*. The solution is then distilled over a water bath and the alcoholic solution of the essential oil collected in the usual manner in a receiver. The temperature of distillation is very important, for with delicate odors, which are of complex composition, there is danger of loss through decomposition. As it is impossible to remove the last traces of oil from the fat, this exhausted pomade is used over again.

Mechanical means for the extraction of the volatile oil from the heavy rinds of citrous fruits, such as the orange, lime, and lemon, are employed.

The nature of the device used varies with the scale of operation. With large quantities, the material is subjected to heavy pressure in a specially constructed press, similar to that used for the extraction of cottonseed oil from cooked seed. For small quantities of material, a shallow metal cup with a hollow handle projecting from the bottom and continuous with the cup is used.

FIG. 25

See Fig. 25. The interior surface of the cup is provided with barbs or spikes, which lacerate the material rubbed on them. The oil thus liberated collects in the hollow handle and is poured off at *a*, Fig. 25.

SYNTHETIC PREPARATION OF PERFUMES

66. Chemical synthesis today furnishes many of the perfumes originally and yet to a large degree obtained from plants. While many of the artificial perfumes have not the intensity of odor of the isolated natural bodies, their greater cheapness guarantees them an eager and enlarging market. In order to present an idea of the methods used in the manufacture of these interesting synthetic products, the preparation of three popular synthetic perfumes will be described.

Great care is required in carrying on the several operations of a single synthesis. Organic reactions, especially with bodies of complex composition, are difficult to control and unless carefully guided are apt to produce bodies widely different from those desired.

Cumarin, a lactone C_9H_6 , $\begin{array}{c} O \\ \diagup \quad \diagdown \\ CH=CH \end{array} - CO$ is the source of

the odor of the tonka bean. It is prepared from 3 parts of salicylic aldehyde, 5 parts of acetaldehyde, and 4 parts of anhydrous sodium acetate. This mixture is heated in a flask for several hours until the reaction is complete. It is then allowed to cool until the mass becomes solid. On the addition of water, an oil separates, which on distillation yields cumarin, which is then crystallized from alcohol.

Heliotropin, an aldehyde $CH_3 \begin{array}{c} O \\ \diagup \quad \diagdown \\ O \end{array} C_8H_7 - COH$ is obtained synthetically from isosafrol, a modified form of safrol; 150 parts of concentrated sulphuric acid, 100 parts of bichromate of potash, and 350 parts of water are mixed and carefully added to 20 parts of isosafrol. The mixture develops more or less heat, which if not carefully regulated greatly influences the character of the resulting product and the yield. The mixture is then distilled over with steam and the distillate purified by extraction with ether. It is further purified by isolating it as a bisulphite compound in virtue of its aldehyde character. The washed

crystals are then decomposed with sodium-carbonate solution. Heliotropin possesses the characteristic odor of heliotrope.

Ionone, a ketone, is the artificial perfume of fresh violets. Equal parts of citral and acetone are mixed with barium-hydrate solution. On completion of the reaction, the mixture is extracted with ether and the residue from the ethereal extract subjected to distillation at a pressure of about 12 millimeters of mercury. The fraction boiling between 138° and 155° C. is further purified by redistillation at the same pressure. The oily distillate is called *pseudo-ionone*. This product is transformed into ionone by heating carefully with dilute sulphuric acid.

COLORING TOILET SOAP

67. General Remarks.—The coloring of toilet soap is a very simple process. If the color is used in the dry state, it is either simply dusted on the soap in the hopper of the mill, spread upon the thin soap while passing between the rollers of the mill, or added to the soap in the box after it has received its preliminary milling. The application of the coloring material in solution is preferable to the dry state, as it admits of a more homogeneous distribution of the color in the shortest time.

There should be a consistency between the nature of the coloring material used, the odor of the perfume, the character of the soap, and the name applied to it.

The *organic coloring materials* commonly used are obtained from coal tar and receive trade names that give no indication to the purchaser of their composition. Red, pink, orange, yellow, green, brown, blue, and lilac colors are obtained already compounded from dealers in perfumers' materials and with guaranteed solubility in definite parts of hot or cold water. The primary qualifications of a soap dye are inertness to the action of alkali and permanency under the conditions in which the soap is used.

TABLE V

COLORS USED IN SOAP AND CHIEF MATERIALS FOR PRODUCING THEM

Color	Material
Red	Coal-tar colors: vermilion, Venetian red, alkanet, bole, colcothar, cinabar, chrome red, carmine.
Orange and yellow..	Coal-tar colors: chrome yellow, cadmium yellow, curcumin, gamboge, turmeric.
Green.....	Coal-tar colors: ultramarine green, chlorophyl extract.
Brown	Coal-tar colors: brown oxides of iron, mixed blue and yellow colors.
Blue and lilac.....	Coal-tar colors: ultramarine blue.

The intensity of the color is determined by the amount of dye used and the character of the soap; coconut oil, for instance, as an ingredient of a toilet-soap base will permit of a greater brilliancy than would be possible without its use.

THE MANUFACTURE OF SOAP POWDER

68. General Remarks.—The manufacture of soap powder in recent years has grown into a business of considerable magnitude. The earliest of those engaged in its manufacture found the business extremely profitable, but with growing competition the margin has been considerably reduced. Well-made, settled, resinous soap can be filled

with upwards of 10 per cent. of soda-ash solution with good results. If this filling be increased in quantity until present in the mixture greatly in excess of the soap, we have the compound which, when reduced to a finely divided state, constitutes the soap or washing powder of trade. The first consideration in the manufacture of soap powder is the composition of the soap base. Any glyceride soap stock is available for this purpose. Rosin, which forms a soft, sticky soap, with great affinity for water, resulting in the formation of lumps in the powder, should not be used, or at best only in small proportion and in combination with a firm tallow base. The prime requisites of a soap powder are freedom from lumps and uniformity of size of the particles composing it. Many soap powders are more finely divided than others, the degree of pulverization being determined by either the fancy of the manufacturer or the demand of the trade.

The manufacture of soap powder is a simple operation, requiring comparatively little outlay for apparatus. As with all manufacturing operations, success is determined not by the ease, simplicity, or cheapness of production, but by the volume of sales of the product. But few proprietary soap powders have a general sale, the great majority of them being confined to a strictly local market.

69. Any settled or semi-boiled non-resinous soap may be used as the soap base. If circumstances do not warrant the separation of the glycerine by graining the soap, a simple semi-boiled soap composed of suitable ingredients will satisfy every requirement. In outlining the process of manufacture, we will begin with a kettle charge of 10,000 pounds of grease. This will require for saponification 10,000 pounds of 20° Baumé caustic-soda lye, yielding in the kettle, by the semi-boiled process, upwards of 20,000 pounds of soap. In killing the stock, follow the directions outlined under the stock change in the manufacture of settled rosin soap. When the stock has been completely saponified and the mass thinned out, or boiled to the proper consistency, it can be crutched at once if the manufacturing

operations demand it. It is best, however, to let it settle and cool over night.

If settled soap which has been grained, strengthened, and then thinned out to the usual consistency is used, it will give the customary yield of 50 per cent. and contain about 30 per cent. of water. With soap made as described above, the yield is 100 per cent. and contains over 45 per cent. of water. This will give us a very liquid soap, to which the soda ash in the dry state can be directly added. With an ordinary settled soap, it will be necessary, depending, however, on the quantity desired to be added, to add a portion of the soda ash in saturated solution. This liquid addition thins out the soap and thereby enables it to incorporate more dry soda ash than it otherwise would.

We have 20,000 pounds of semi-boiled soap; to make a soap powder containing 2 parts of soda ash to 1 part of soap (which proportion will yield a good powder), it is necessary to add 40,000 pounds of soda ash, giving a yield of 60,000 pounds of soap powder from 10,000 pounds of grease.

70. Crutching.—Pump the crutcher one-third full of soap and start it, adding gradually the requisite quantity of soda ash, which has been previously weighed out. When the mixture has been made homogeneous, it is dropped into the frame and slabbed as soon as it has cooled sufficiently to permit the removal of the frame sides. The slabs are cut into narrow bars of a length corresponding to the width of the frame. These bars are then racked and dried. Soap and soda ash are generally mixed in large quantities at a time and reduced to powder as required. When sold in quantities, it is usually shipped in barrels; when intended for the retail trade, it is packed in cartons marked with the brand and name of the manufacturer.

71. Soap-Powder Mill.—The soap-powder mill is of especial importance and should be so constructed as to do the work quickly, without heating the powder, and with a minimum of dust and repairs. The machine shown in Fig. 26 receives the bars of soap powder, previously broken

into lumps, in a hopper attached to the collar *a*, and crushes, grinds, and sifts them in one continuous automatic operation. The lumps are reduced by repeated blows of rapidly revolving steel blades, or beaters, enclosed in the semi-cylindrical casing shown at *b*. The powder is discharged

FIG 26

through a semi-cylindrical screen, which serves as a sifter, and which forms the under side of the casing *b*. Power is applied by belt at *c*. The mill is mounted upon a well-braced framework completely enclosed to retain dust and high enough to admit the introduction of a barrel to receive the powdered material.

THE RECOVERY OF GLYCERINE FROM WASTE SOAP LYE

THE MANUFACTURE OF GLYCERINE

INTRODUCTORY

72. Historical.—Scheele, in 1779, when preparing lead plaster by heating olive oil with litharge, obtained a soluble, sweet-tasting substance, and later, in 1784, he found that the same substance could be obtained from other oils, as well as from butter and lard. To this material he gave the name "the sweet principle of fats," and it afterwards bore the name of Scheele's sweet principle, or oil sugar.

Lead plaster is said to have been discovered by the Roman physician Menecrates about the middle of the 1st century, and also to have been known to Pliny, who briefly described its uses, mode of preparation, and application, but nothing was known of *glycerine* until Scheele's day.

Later the body was more carefully investigated by Chevreul, who determined its composition with tolerable exactitude and gave to it the name which it now bears. Pilouze in 1836 first established its formula. His experimental results corroborated Chevreul's views that the fats are ether-like compounds of the fatty acids. Henceforth glycerine became the subject of study by Berzelius, Liebig, Berthelot, and de Luca; but it remained for Wurtz to determine its exact chemical composition and relation to other bodies of the aliphatic series.

Scheele published the results of his investigations in a communication which appeared in the Transactions of the Royal Academy of Sweden, in 1783.

He describes his method of preparation in the following terms: "It is not generally known that all solid oils obtained by pressure contain a natural sweet principle which differs in its special relations and properties from the other well-known saccharine materials occurring in the vegetable kingdom. This sweet principle makes its appearance when oils of this kind are boiled with litharge and water until the whole of the litharge is dissolved by the oil. Water is then poured upon the 'emplastrum simplex' thus formed, the whole boiled for a few minutes, and on cooling the liquid is filtered off from the plaster and boiled until the residue becomes sirupy."

73. Glycerine was prepared by this process alone for many years, the lead introduced as an impurity from the litharge being removed, before concentrating the filtrate, by the use of hydrogen sulphide. This concentrated filtrate, after some primitive clarification, constituted the glycerine of commerce. Its rapidly increasing use soon demanded its production upon a larger scale. It was known that in the

process of soap making the glycerine, liberated from the fat in the act of saponification, remained in the waste lye, but efforts to recover it from this medium with the extremely crude methods used were unsuccessful. In the meantime the manufacture of stearin candles was undergoing important developments. Numerous patents were granted for processes for the decomposition of the fats and the separation of glycerine. Gay-Lussac and Chevreul, in 1825, were the first to obtain patents. Their patent was for the alkaline saponification process, which, however, did not admit of successful operation until after the improvement by de Milly, in 1831. This is the autoclave process at present in use.

During the succeeding 20 years the sulphuric-acid distillation process was proposed and developed. Although other investigators had worked with the problem, and in fact a form of the process was in actual use by the predecessor of the Price Patent Candle Company, it remained for an American chemist, R. A. Tilghman, to determine the conditions productive of the most successful results. For his originality he received a patent in 1854, having previously filed a caveat. An English patent was soon after taken out. Tilghman's process is that at present employed in conjunction with the use of sulphuric acid and is described in Part 1.

74. In 1847 Sobrero in Paris discovered **nitroglycerin**. Alfred Nobel demonstrated its value as an explosive in 1863 and in 1866 invented dynamite. This was followed in 1875 by blasting gelatine. The construction of vast engineering undertakings was greatly facilitated by the use of these substances, of which glycerine is the basis. The increased use reacted directly upon the source of the raw material, and the soap trade soon came to a perplexing realization of the increasing value of the product being daily consigned to the sewer.

75. Treatment of Waste Lye.—In the discussion of the manufacture of settled resinous soap in Part 1, we traced the progress of a boil of soap with the formation of the various lyes. These are dilute solutions of varying percentages

of sodium hydrate, sodium carbonate, sodium sulphate, sodium chloride, and glycerine, contaminated by more or less soap in suspension, together with coloring matter introduced by the rosin, and with some mucilaginous matter or animal tissue remaining with the stock when rendered. This often foul-smelling liquor, a by-product with the soap maker, now becomes the raw material of the glycerine refiner.

The process and apparatus in most general use in this country and abroad represent the culmination, in efficiency and simplicity, of all systems of glycerine recovery from waste lye. It has been declared impossible to prepare a chemically pure glycerine from waste lye—that glycerine of dynamite grade represents the limit in purity of glycerine obtained from this source. Soap lye, by the improved treatment at present in use, is made to yield a glycerine of the highest purity and answering the severest requirement of any pharmacopœia. In the process that has received the substantial approval of general practical use, the agents employed for the precipitation of albuminous and soapy matter and the neutralization of the alkali present in the lye are oil of vitriol and a basic sulphate of iron.

The latter body is prepared by treating pulverized iron ore with sulphuric acid, whereby an acid salt, mixed with some uncombined ore and acid, is obtained. This mixture is subsequently subjected to a high temperature, viz., 380° to 500° F., under which condition it undergoes a transformation into a basic sulphate of iron. The efficiency of this body as a clarifying agent depends on the formation of ferric hydrate and insoluble iron soaps that entangle the albuminous matters, all of which settling through the liquor clarify and decolorize it.

76. In the glycerine refinery, the waste lye as it is received is usually elevated to the top of the building, where it is reserved in storage until required for use. It is lowered by gravity to the mixing tank, where it receives the proper proportions of sulphate, previously dissolved in hot water,

and vitriol. The sulphate, in virtue of some free acid that it contains, acts also as a neutralizing agent. Besides a quantity of sulphate sufficient to effect the clarification desired, vitriol is added, for the sake of economy, to complete the neutralization. After this preliminary treatment, the lye is filtered by means of a filter press and is ready for the first evaporation.

The lye may undergo other treatment, as, for instance, a repeated precipitation, to render more effective and complete the clarification and decolorization.

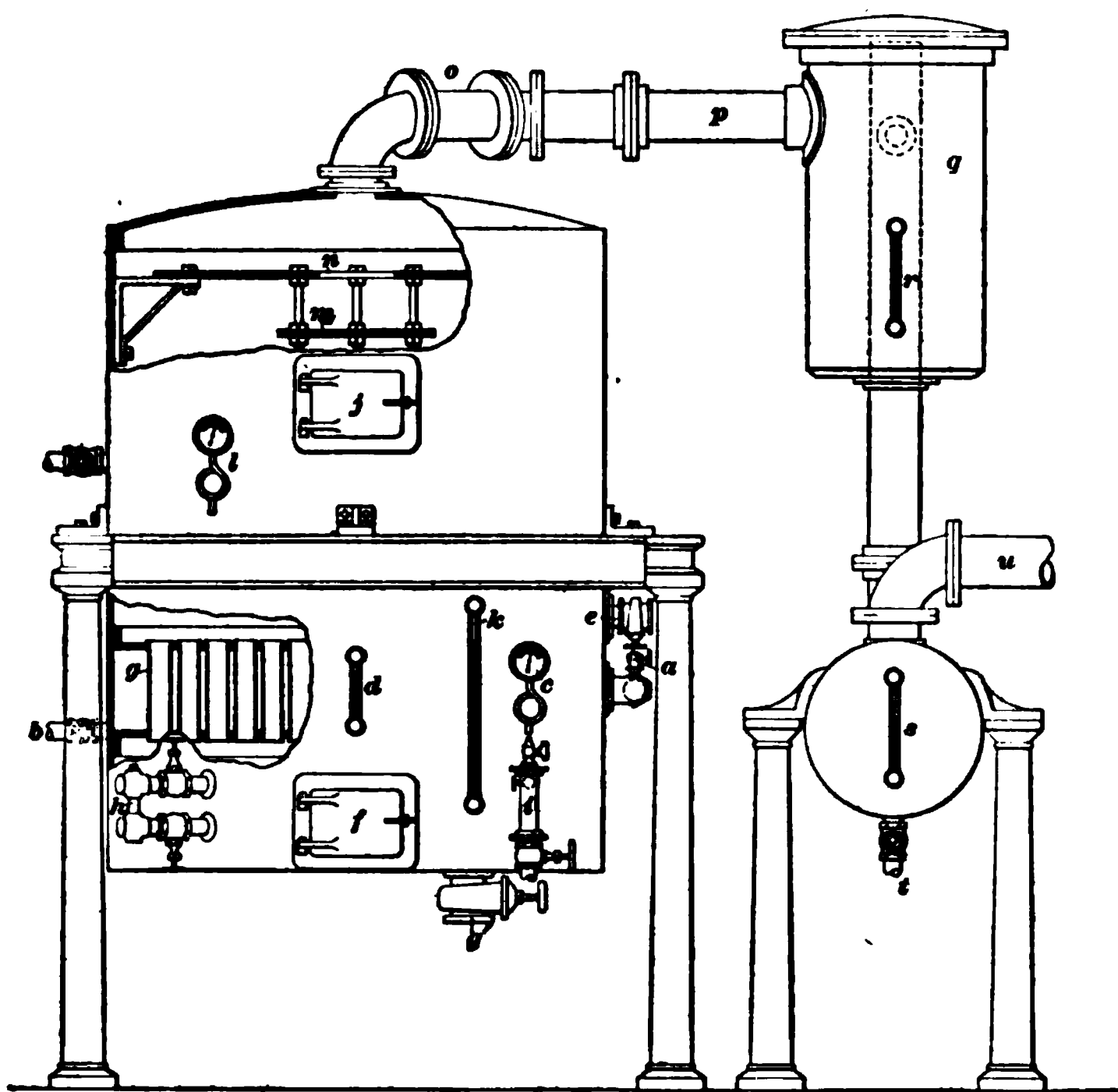


FIG. 27

77. Evaporation of the Lye.—The purified lye is now drawn by vacuum into the lye evaporator shown in Fig. 27.

This consists of cylindrical tanks of sheet steel supported on pillars and provided with pipes for the removal of vapor,

entrance and exit of steam, introduction of fresh lye, and with manholes for cleaning and removal of salt and evaporated lye. They are also provided with gauges for the indication of vacuum, steam pressure, and level of liquor, and a device for the collection of samples. Evaporation is effected by use of exhaust or live steam, which is admitted into a chamber filled with vertical tubes through which the lye passes. Intimate contact with the source of heat is thus obtained. A vacuum of about 28 inches is maintained with the regulation wet-air vacuum pump. The pipes through which the vapors pass from the boiling liquor connect with catch-alls for the collection of entrained matter, and these in turn communicate with the vacuum pump.

In this evaporator, Fig. 27, *a* is the pipe for the entrance of live or exhaust steam into the steam chest shown in section at *g*; *b* is the outlet for the water of condensation from the steam chest, the level of which is shown by the gauge glass *d*. The pipe *e* is for the introduction of the liquor to be evaporated. The door *f* is for the removal of the salt. The pipe *g'* is for the removal of the concentrated liquor. The pipes *h* are to enable the attendant to discharge the concentrated liquor close to the level of the salt. The device *i* is for the removal of samples. The door *j* is to permit an examination of the interior of the evaporator. A gauge glass *k* shows the level of liquor in the evaporator. The vacuum gauge *l* shows the vacuum inside the evaporator. The baffle plates *m* and *n* prevent the entrainment of liquor through the pipes *o* and *p* into the catch-all *q*, which is provided with the gauge glass *r*, to indicate the level of any liquor therein. A horizontal cylinder *s* collects the entrained matter, which may be removed through *t*. The pipe *u* leads to the vacuum pump by means of which a vacuum is maintained in the system.

78. Evaporation is continued in the lye evaporator until a concentration of over 26° Baumé is indicated. The vacuum is then broken through the pipe *u* and the concentrated lye discharged through the pipe *g'*. The door *f* is

then opened and the lye allowed to run upon a filter, upon which the crude salt is afterwards scraped. Suction is applied to the wet salt and the lye is thoroughly removed. The chief mineral constituent is $Na_2SO_4 + 12H_2O$, or Glauber's salt. The recovered salt is washed to eliminate the last traces of glycerine, dried by suction, and returned to the kettle room to be used in graining soap, thus completing the circle. The 26° Baumé lye, as it is now called, is a saturated salt solution. The percentage of glycerol is generally about twice as great as that of the fresh lye, the volume of the latter having been reduced practically one-half.

79. The object is to remove as much salt as possible from the waste lye concentrated in the evaporator shown in Fig. 27. The more salt there is separated here, the less remains to be removed in the subsequent evaporation. The concentrated lye is now subjected to a second and final evaporation, whereby crude glycerine is produced. This may be effected in the same evaporator, but in large plants a smaller evaporator of precisely the same construction is used. The separation of the salt from the crude glycerine is also effected in the same manner as described above.

TREATMENT OF CRUDE GLYCERINE

80. Crude glycerine contains from 75 to 85 per cent. of glycerol, about 6 per cent. of salts, chiefly sodium chloride, about 5 per cent. of organic matter, constituting the foots obtained on subsequent distillation, while the remainder is water. It is a thick, viscous liquid, its color and clearness depending in a great measure on the efficiency of the preliminary treatment that the fresh lyes have received for the removal of organic matter, including iron salts of the organic acids.

81. Distillation of Crude Glycerine.—The crude glycerine is withdrawn into storage tanks from which it is

transferred, as required, to another vacuum apparatus in which the distillation is conducted. This apparatus comprises a large cylindrical sheet-iron tank, in which the crude glycerine is distilled and a series of vertical pipes (terminating at the bottoms in horizontal cylindrical reservoirs) in which the distillate condensed in the vertical pipes collects. These are termed catch-alls, and are usually from three to five in number. At the end of this series of receivers are two larger horizontal cylindrical vessels surmounted by an iron cylinder filled with water-cooled tubes, through which the more volatile portion of the distillate passes and is condensed, falling into the reservoirs below. These vessels, called *sweet-water drums*, communicate with a dry-air vacuum pump, which maintains a vacuum of about 28.5 inches throughout the system.

As a rule, there are two of these stills, with their accompanying condensers and receivers, the same pump maintaining a vacuum in both. The distilling vessel is of various sizes, depending on the capacity of the plant. It is a well-made, upright sheet-iron cylinder about 5 feet in diameter and about 10 feet in height. Steam at 125 pounds pressure, corresponding to a temperature of 345° F., circulates in a closed coil and maintains a constant temperature.

82. Advantage of System.—The essential advantage of this system of glycerine distillation consists in the injection of expanded and reheated steam into the body of crude glycerine maintained at a high temperature. Glycerine has a boiling point of 290° C., or 554° F. Under atmospheric pressure, however, it cannot be heated to this temperature without undergoing decomposition. By reducing the pressure, as is done in a closed vessel when a vacuum is maintained, it boils without decomposition, and its vapor may be condensed unchanged. We have in commercial crude glycerine a very impure body, the organic matter present causing difficulty in the refining process. To effectually separate this organic matter, recourse must be had to some

means other than boiling at a lower temperature under reduced pressure. To accomplish this, a jet of expanded and reheated steam is introduced into the mass of crude glycerine heated to the temperature of distillation.

If steam at boiler pressure, viz., 125 pounds, were used directly for distillation without previously being expanded and reheated, its expansion in the distilling apparatus would not only greatly retard, but would practically stop the operation. To the great loss in heat due to absorption by the expanded steam would be added the difficulty arising from its condensation. These adverse and prohibitory conditions are eliminated by the use of expanded and reheated steam, which is prepared in the following manner: Steam taken directly from the boilers is allowed to expand in a coiled pipe of larger diameter contained in a well-insulated vessel, called a "reheater," which is attached to each still and in many cases is larger than the still itself.

The steam in expanding suffers a reduction in temperature. The heat lost is restored by surrounding the coil within the reheater with the same unexpanded steam from the boiler. Steam thus treated is introduced directly into the crude glycerine, which distils without decomposition at a temperature of about 300° F. Before introducing the steam, crude glycerine is added in sufficient quantity to cover the jet pipe to a satisfactory height, as indicated by the gauge glass, and as the distillation continues, crude glycerine is added from time to time in quantities not too great to interfere with the even working of the still; never, however, allowing it to fall below the established level. If, from lack of attention on the part of the attendant, an excessive quantity is introduced, there is great danger of the entrainment of the impurities contained in the crude glycerine, which increase in quantity in the still as the distillation proceeds.

83. Crude-Glycerine Still.—Referring to the still shown in Fig. 28, *A* is the still proper, *B* the reheater, *C*

and *C'* the catch-all drums, and *D* the sweet-water drum. In the still *A*, the closed coil, shown in dotted lines, carries

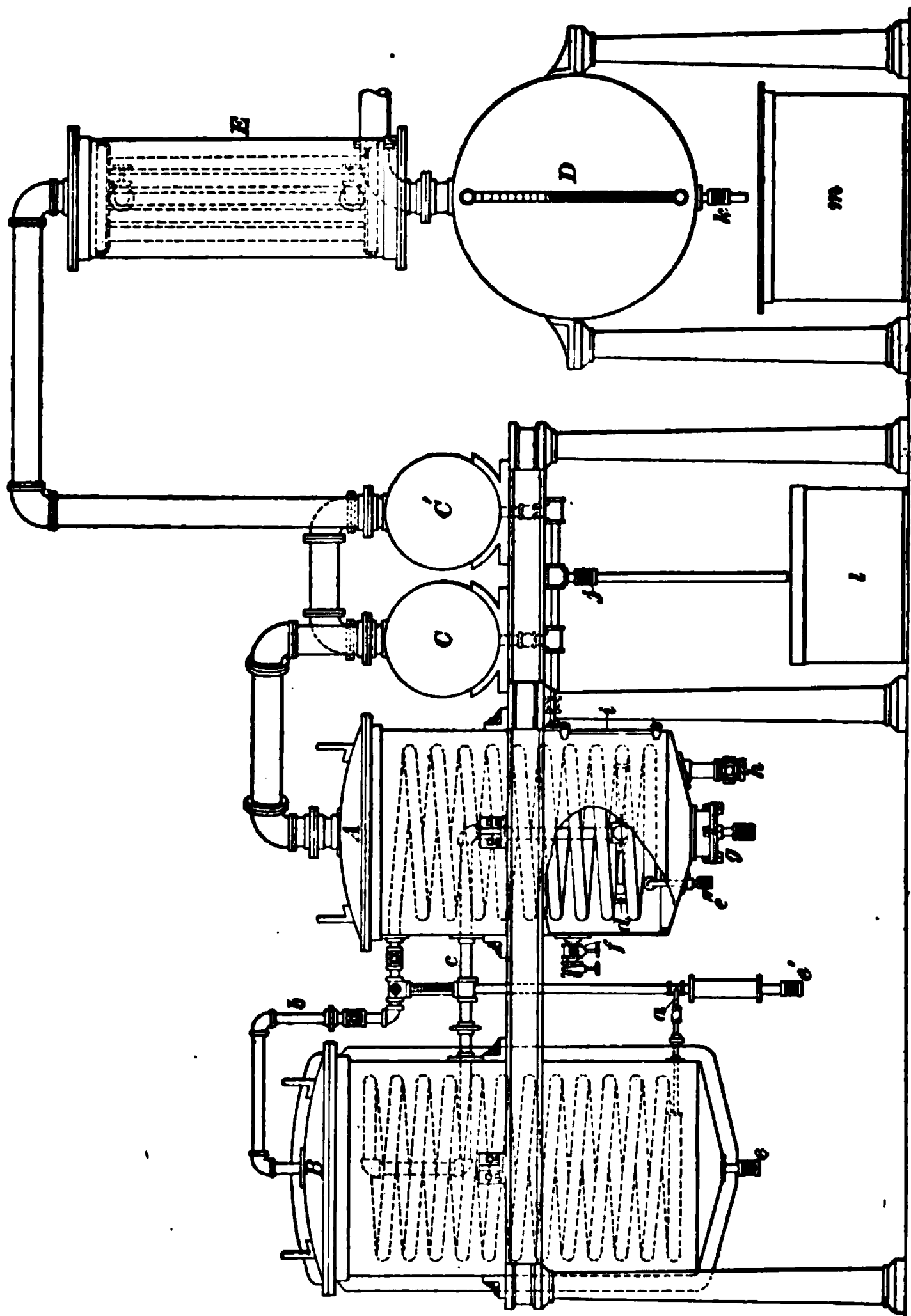


FIG. 28

steam at boiler pressure, which heats the contents of the still to the temperature required for distillation. In the

reheater *B*, steam at 125 pounds pressure enters through the pipe *a* and expands into the coil of larger diameter, which is shown in dotted lines. This expansion coil is surrounded by steam at boiler pressure entering the reheater through the pipe *b*, which also carries steam at the same pressure into the reheating coil of the still *A*. Expanded and reheated steam from the reheater enters the still through the pipe *c* and discharges into the crude glycerine contained therein through the crisscross jet shown at *d*. The catch-all stuff, so called, collects in the catch-all drums *C* and *C'*, and the lighter portion of the distillate condensed in the water-cooled jacket at *E* collects in the sweet-water drum *D*. At *e*, *e'*, and *e''* are outlets for the water of condensation from, respectively, the reheater *B*, the high-pressure steam line from the boiler, and the heating coil of the still *A*. At *f*, crude glycerine is fed into the still by suction. At *g* is the drop door through which the "foots" in the still *A* is discharged. Water used in washing the still is discharged at *h*. The level of the contents of the still is shown by the gauge glass *i*. At *j* and *k* are the discharge valves for the contents, respectively, of the catch-all drums *C* and *C'* and the sweet-water drum *D*. Tanks *l* and *m* are reservoirs for these respective portions of the distillate. All steam pipes leading to and from the apparatus, as well as the still and reheater, are insulated to retain the heat.

Before beginning a distillation, it is customary to have in stock a considerable quantity of crude glycerine, so that there will arise no necessity of shutting down the apparatus before conditions in the still render it imperative. Fresh lye is undergoing concentration all the time, and naturally the product of one operation comes in greatly reduced bulk to be the raw material of the next.

84. Products of Distillation.—The products of the distillation of crude glycerine may be separated into three classes, viz., material collected in the catch-alls, technically

called *half refined*, from its degree of purification; material of a much lighter gravity, called *sweet water*, consisting chiefly of water containing from no glycerine at all to as high as 12 per cent.; and a black, viscous residuum left in the still constituting the *foots*.

85. The **half-refined glycerine** that is collected in the catch-all drums *C* and *C'*, Fig. 28, is of a bright straw color. This product undergoes a second distillation, whereby its purity is greatly increased. After the second distillation it undergoes a concentration to expel all water, whereupon it is run into drums and is ready for the market.

The **sweet water** is concentrated to the consistency of crude glycerine and is then distilled and the distillate concentrated, as explained for half-refined glycerine.

86. Foots.—There remains to be considered the black residuum in the crude glycerine still. All the organic matter not removed in the preliminary treatment of the lye accumulates in the still and forms this black, viscous residue, which, if the distillation be carried too far, becomes so hard as to be removed from the still only with great difficulty. To facilitate its easy handling and the recovery of the considerable quantity of glycerine retained by it, the distillation is checked at that stage beyond which there is danger of contaminating the distillate with entrained matter from the still. The quantity of glycerine present depends on the concentration and is about 50 per cent.

In the distillation of crude glycerine the organic compounds of sodium do not precipitate as do the inorganic compounds, viz., sodium sulphate and sodium chloride, and the former, as the mass in the still undergoes concentration, remain to constitute the **foots**. There are present, usually, about 5 per cent. of salt, 20 to 30 per cent. of sodium acetate, and from 10 to 15 per cent. of sodium salts of non-volatile organic acids.

While yet soft and comparatively fluid, the foots is transferred to a tank and neutralized with oil of vitriol. After the gradual addition of the diluted acid and filtering, the product is concentrated, forming crude glycerine from foots; this is then distilled. About 1,200 pounds of foots are obtained from every 10,000 pounds of crude glycerine, yielding on distillation about 50 per cent. of glycerine.

The glycerine obtained by the process described constitutes the dynamite glycerine of commerce. It is of a pale straw color and requires only subsequent distillation and filtration through bone black to become the water-white, chemically pure glycerine of the pharmacopœia. Its specific gravity is an important physical characteristic, and is subject to considerable variation. A density of 1.263 at 15°C. is commonly obtained.

A sample from each run of refined glycerine is analyzed for sodium chloride, ash, carbonaceous residue on ignition, and for acidity, both free and combined, in addition to a determination of the specific gravity. Samples, on standing, tend to accumulate color, due doubtless to an oxidation process of a nature not clearly understood.

87. The chemical examination of the product, not only during the various stages of the refining process, but also at its completion, is a matter of the utmost importance. By this close espionage the refiner is warned of changes too delicate for observation, and is thus able to correct in its first stages any abnormal behavior that would deteriorate the final product.

The examination of the refined glycerine not only indicates the care exercised in the refining, but forms the basis for the valuation and sale. The influence exerted by foreign matter in the glycerine upon its subsequent use in nitration and the manufacture of dynamite demands a searching chemical and physical examination that is entirely warranted by the danger attending these processes.

CHEMICAL EXAMINATION OF RAW MATERIALS AND PRODUCTS

INTRODUCTORY

88. Importance of Chemical Examination.—Every large soap manufactory maintains a chemical laboratory and employs a trained chemist whose duty it is to examine all raw materials, thereby enabling the purchasing department to avoid losses incident to misrepresentation, and to insist upon a high grade of material being offered. The chemist analyzes soap and similar products made by competitors to obtain the necessary information whereby his employer is enabled to promptly duplicate it, if desired, at the same or a lower figure. He examines daily the various products that occur in routine factory work and promptly informs the superintendent of conditions requiring modification or elimination. Glycerine recovery cannot be carried on with complete satisfaction without some knowledge of the chemical principles involved in its formation in soap manufacture and its recovery from waste soap lye. Refined glycerine is sold under a guarantee of its quality, to determine which skill in chemical analysis is necessary. As soap manufactories grow in size and as competition increases, the services of a chemist become more indispensable. It may be stated that the development of the soap industry to its present proportions would not have been possible without the aid of the chemist.

In *Quantitative Analysis* is given a thorough discussion of the analysis of fats, waxes, and mineral oils, and also a general resumé of the subject of soap analysis.

The discussion at this time of analytical methods employed in the soap-works laboratory is supplementary to the information given in the above mentioned Paper.

89. In industrial laboratory work, owing to the quickness with which the information obtained by chemical

analysis is desired, many of the analytical methods given in technical treatises are seldom or never used, or only in part. In the routine laboratory work, absolute accuracy, while desired, is not required in every case, it being sufficient that successive determinations are performed as accurately as the time will permit and that they be uniform, for with uniformity of manufacturing operations, the analytical results must be on a perfectly comparative basis.

EXAMINATION OF SOAP STOCK

90. Collection of Sample.—Remove the bung from the cask, and with the long steel sampler remove portions from the cask in as many different directions as desired to obtain a representative sample and of suitable amount. If different portions of the sample removed from the cask vary considerably, it will be necessary to melt the entire sample to insure homogeneity of composition. If uniform throughout, different portions of the sample may be taken and used for all of the determinations. In warm weather, incipient fusion of the sample is unnecessary. In cold weather, it need be applied only to tallow and firm grease, coconut oil and cottonseed oil being easily and quickly sampled.

91. Determination of Moisture.—Weigh into a weighed 2½-inch porcelain evaporating dish from 3 to 5 grams of the well-mixed sample. Heat in a drying oven, maintained at a constant temperature of 105° C., for 1 hour. Cool and weigh. The loss in weight multiplied by 100 and divided by the weight of the sample taken will give the percentage of moisture. This percentage is always appreciable in animal soap stock, and in the contract purchase of large quantities, rebate is generally allowed for a moisture content in excess of an allowed percentage. Cottonseed oil ordinarily contains no moisture, and the moisture determination, if applied, will generally show an increase in weight owing to oxidation.

With coconut oil, the volatile matter is frequently, if not invariably, in excess of the actual moisture content, owing to the volatility of the fatty acids of low molecular weight.

92. Determination of Free Fatty Acids.—Aside from the melting point and titer, this is the most important test applied to soap stock, as it gives not only an indication of the value of the stock as a source of glycerine, but of the care exercised in its manufacture and of its origin. The percentage of free fatty acids in soap stock varies considerably from season to season, being greater in the summer than in the winter, owing to the fact that the agents of decomposition are more active in the former season. This is shown in the following tabulation of the analytical data resulting from a long series of tests applied to the stocks mentioned:

Soap Stock	Percentage Free Fatty Acids	
	Summer	Winter
Tallow	7.85	5.85
Bone stock.....	10.68	6.64
Grease.....	18.03	7.49

As about 10 per cent. of glycerine is theoretically available from these soap stocks, a free fatty acidity of 10 per cent. will indicate a loss of 1 per cent. of glycerine, calculated on the basis of the stock, or 10 per cent. calculated on the amount of glycerine theoretically available.

93. The method of procedure is as follows: Heat the sample sufficiently to melt it and transfer 5 cubic centimeters to a wide mouth Erlenmeyer flask of 250 cubic centimeters capacity, weigh, and add about 50 cubic centimeters of

95-per-cent. alcohol (neutral). Insert in the mouth of the flask a cork carrying a long glass tube to serve as an air-return flow condenser, and heat the contents of the flask on a steam bath for 5 minutes.

The free-fatty-acid determination, with alcohol as a solvent, is based upon the practical insolubility of neutral glycerides in alcohol, while the free fatty acids are soluble. Remove the condenser, use phenol phthalein as indicator and titrate with semi-normal caustic soda. Calculate free fatty acids in terms of oleic acid whose molecular weight is 282. One cubic centimeter of $\frac{n}{2}NaOH = .141$ gram of oleic acid. Assuming that x cubic centimeters of $\frac{n}{2}NaOH$ are required for neutralization, the percentage of free fatty acids may be found according to the following formula:

$$\text{Percentage of } \left. \begin{array}{l} \text{free fatty acids} \end{array} \right\} = \frac{x \text{ c. c. } \frac{n}{2} NaOH \times .141 \times 100}{\text{weight taken}}.$$

By measuring a definite volume of the liquid fat much time is saved, it being a simple matter to ascertain the average weight of 5 cubic centimeters of the soap stock customarily examined.

94. Determination of Melting Point. — The term **melting point**, as usually employed, indicates the temperature at which the portion of fat examined becomes transparent. This temperature, however, varies under different conditions, depending chiefly on the initial temperature of the body, the time during which the temperature of the fat is raised to transparency, and the consistency, or composition, of the fat. The melting point of a fat is considered to be that temperature at which a thin disk of fat, when freed from the attraction of gravitation and left to its own molecular forces, assumes a globular state. Where a large number of determinations are made daily, a special melting-point apparatus is used.

95. Melting-Point Apparatus. —The essential parts of a melting-point apparatus are shown in Fig. 29. It consists

of a large test tube *e*, about $1\frac{1}{2}$ inches in diameter and 5 inches long, which is filled first with distilled water to one-third of its height. Absolute alcohol is now added slowly, to avoid mixing, in quantity sufficient to fill another third of the tube. The test tube is supported in a large, round-bottomed glass beaker *d* filled with water to serve as a water bath and supported from the stand *f* and heated with a Bunsen burner. The water in the beaker is kept in agitation by means of an air jet furnished by bulb *g*, whereby water at an equable temperature surrounds the glass tube. The thermometer *c* is for reading the temperature of the bath.

FIG. 29

A disk of fat, from 1 to 1.5 centimeters in diameter, prepared by allowing a drop of molten fat to fall from the end of a glass rod on a flat piece of ice, is dropped into the test tube. Being of greater density than alcohol and lighter than water, it remains suspended at about the line of demarcation of the two liquids. A thermometer *a*, divided into tenths of a degree, is suspended in the test tube, with its bulb beside the disk of fat. As the temperature slowly rises, the

consistency of the fat is observed. The temperature indicated by the thermometer is read off by the aid of a magnifying glass when the disk assumes a globular shape free from turbidity. Uniformity of treatment in successive determinations is essential. A different melting point can be obtained with the same sample by failure to observe uniform conditions.

Where the determinations of melting point are numerous, several of these apparatus may be arranged in a row, thus allowing a number of determinations to be carried on at once.

The percentages of moisture and of free fatty acids and the melting point, together with the simple but invaluable sense tests of odor, grain, and color, amply suffice to gauge the quality of any sample of soap stock. When suspicion is aroused by the generally inferior appearance of any sample, it is well to apply the tests for unsaponifiable matter and for the percentage of alkali absorbed.

96. Determination of Alkali Absorbed.—Weigh 2 or 3 grams of the sample into an Erlenmeyer flask of the same size as was used in the free-fatty-acid determination. Run in from a burette an accurately measured quantity of alcoholic potash of known strength sufficient to saponify the weight taken and to leave some excess. Heat on a steam bath with an air condenser until the soap formed has completely dissolved. Neutralize the excess of caustic potash required for saponification with $\frac{n}{2}H_2SO_4$, using phenolphthalein as indicator. Always run a blank determination. From the difference in the number of cubic centimeters of semi-normal acid used for the blank determination and the real determination, the amount of sodium hydrate required for saponification can be calculated.

ILLUSTRATION.—The determination of the percentage of sodium hydrate absorbed will be evident from the following calculation, in which 25 cubic centimeters of alcoholic potash is used:

1 cubic centimeter of alcoholic potash equals .8256 cubic centimeter of $\frac{n}{2}H_2SO_4$; 25 cubic centimeters equal 20.64 cubic centimeters of $\frac{n}{2}H_2SO_4$.

The amount of $\frac{n}{2}H_2SO_4$ required to neutralize the excess of alcoholic potash is 1.87 cubic centimeters; the amount of alcoholic potash, in terms of $\frac{n}{2}H_2SO_4$, required for saponification is 19.27 cubic centimeters.

1 cubic centimeter of $\frac{n}{2}H_2SO_4$ equals .02 gram of sodium hydrate.

$19.27 \times .02 = .3854$ gram of sodium hydrate absorbed by fat.

$\frac{.3854 \times 100}{\text{weight taken}} = \text{per cent. of sodium hydrate absorbed.}$

By reference to Tables XI and XII, Part 1, it can be ascertained whether this percentage is normal or abnormal. If the percentage of sodium hydrate absorbed is below the amount usually required by stock of this character, the nature of the inert material present demands examination.

97. Determination of Unsaponifiable Matter.—Evaporate the contents of the Erlenmeyer flask of the preceding determination to perfect dryness and extract the residue with naphtha on a water bath, using a return-flow air condenser. When the extraction is complete, filter contents of flask into a weighed beaker and evaporate the solvent and weigh the residue. This will give us the percentage of unsaponifiable matter present in the stock and soluble in the menstruum used.

If lime soap be present, this may be ascertained by treating the stock with dilute sulphuric acid, whereupon a precipitate of calcium sulphate is obtained, which can be confirmed by the ammonium-oxalate test. The percentage of foreign matter, animal tissue, etc. is determined by extracting a weighed portion of the sample with naphtha, whereupon all the fat dissolves. The solution is filtered through a weighed filter which, after having been washed with naphtha free from all traces of fat, is weighed. From this the percentage of foreign matter, etc. present in the stock can be calculated.

98. Titer Test.—All soap stock of animal origin is bought and sold on the "titer," by which is meant the highest temperature produced by the latent heat of fusion that is liberated on the solidification of the pure liquid fatty acids

obtained from the stock. This test is also called the hardness test.

In *Quantitative Analysis* is given the method of titer determination as proposed by Wolfbauer. To obtain reliable results by whichever modification of this method that is used, it is essential that each determination be made under precisely uniform conditions and from fatty acids prepared in the same manner. This determination may also be applied to cottonseed and coconut oils. For the former stock, it is a reliable index of the soap-making qualities of different samples of oil. The titer test is time consuming.

ANALYSIS OF ROSIN

99. Determination of Unsaponifiable Matter.—The only chemical test commonly applied to rosin is the determination of the unsaponifiable matter, which increases, as we have learned, as the quality of the rosin deteriorates. The grading accorded the barrel from which the sample is taken is determined by comparing with standard cubes a cube cut from the sample, with dimensions equal to those of the standard. Care must be taken to have the sample cube representative of the quality of rosin in the barrel. The unsaponifiable matter is determined in the same manner as the unsaponifiable matter in glyceride stock.

100. Comparison With Standards.—Rosin of a given grade should correspond in color and clearness to the color and clearness of the standard cube of the same grade. The standards should be removed from time to time as they tend to bleach on exposure and to lose the cube shape through softening. In comparing samples with the standards, strict uniformity of dimensions looked through and exposure to light must be observed.

ANALYSIS OF SODA ASH

101. Determination of Sodium Carbonate.—Weigh 1 or 2 grams of the sample into a 500-cubic-centimeter flask and dissolve in water; add a few drops of phenol phthalein

as indicator. Run in from a burette an accurately measured excess of semi-normal sulphuric acid and boil to expel all traces of carbon dioxide. Titrate excess of acid with semi-normal caustic soda. The solutions are equal and are known in terms of the various sodium compounds. The weight of sodium carbonate corresponding to the number of cubic centimeters of semi-normal sulphuric acid required to neutralize the soda ash multiplied by 100 is divided by the weight of alkali taken, which gives the percentage of actual anhydrous sodium carbonate present in the soda ash.

102. Determination of Total Alkali. — Dissolve 10 grams of the soda ash in about 150 cubic centimeters of warm distilled water and dilute to 1 liter. Remove 50 cubic centimeters of this solution to a beaker and titrate with semi-normal sulphuric acid, using methyl orange as indicator. As 1 cubic centimeter of semi-normal acid is equivalent to .03082 gram of Na_2O , the percentage of Na_2O in the soda ash will be found by multiplying the number of cubic centimeters of semi-normal acid required by .03082 by 20 and by 100 and dividing the result by 10 (the weight of the sample taken).

ANALYSIS OF COMMERCIAL CAUSTIC SODA

103. Determination of Total Alkali.—Proceed exactly as explained for the determination of the total alkali of soda ash. Express the percentage of total alkali in terms of sodium oxide.

104. Determination of Sodium Hydrate.—Weigh out a portion, as previously explained, in a 100-cubic-centimeter graduated flask and precipitate the sodium carbonate with boiling $BaCl_2$ solution. Make up to mark, shake well, and allow flask to stand until the precipitate of barium carbonate has subsided. Filter supernatant liquid and titrate 10 cubic centimeters with semi-normal hydrochloric acid. Semi-normal sulphuric acid cannot be used, as barium sulphate is insoluble. The percentage of Na_2O is found by multiplying

the number of cubic centimeters of semi-normal acid found by $.03082 \times 10 \times 100$, and dividing the result by the weight of entire sample taken.

105. Determination of Sodium Chloride. — Weigh 2 or 3 grams in a 100-cubic-centimeter, graduated flask, dissolve in water, and make up to mark. Transfer 10 cubic centimeters to a porcelain dish 4 inches in diameter. Dilute the aliquot portion taken with water, neutralize with dilute nitric acid and sodium-carbonate solution, using phenol phthalein as indicator and boil to expel carbon dioxide. When perfectly neutral add a few drops of potassium chromate as indicator and titrate with deci-normal silver-nitrate solution to the appearance of a permanent, faint, reddish coloration. Each cubic centimeter of the silver-nitrate solution contains .016868 gram of silver nitrate, which will precipitate .05806 gram of sodium chloride in accordance with the following proportion:

$$\begin{aligned} AgNO_3 : NaCl \} &= .016868 : x. \\ 168.68 : 58.06 \end{aligned}$$

$$x = .005806.$$

Multiply the number of cubic centimeters of silver-nitrate solution used by its equivalent in sodium chloride and the result by 10×100 and divide the whole by the weight of material taken. The result is the percentage of sodium chloride.

The results of these determinations may be expressed as follows (using the figures of an old analysis):

Total alkali as Na_2O	69.55%
Free alkali as Na_2O	61.72%
Combined alkali as Na_2O (by difference)	<u>7.83%</u>
Sodium chloride, $NaCl$	9.40%

To express the complete results in finished form, it is necessary to transform the free alkali, as Na_2O , into free alkali, as $NaOH$, and the combined alkali, as Na_2O , into combined alkali, as Na_2CO_3 ; which done we have the following report:

Total alkali Na_2O	69.55%
Caustic alkali $NaOH$	79.64%
Carbonated alkali Na_2CO_3	10.92%
Sodium chloride $NaCl$	9.40%
Undetermined.....	.04%
Total.....	100.00%

To judge from this report, the commercial caustic soda analyzed is of practically 70-per-cent. quality.

106. Caustic potash and pearl ash are examined in the same manner. If the quantity of potash purchased warrants the work, the percentage of caustic soda, sodium carbonate, potassium chlorides, and the alkali sulphates may be determined. These compounds are present as impurities.

ROUTINE CHEMICAL EXAMINATION OF KETTLE-ROOM PRODUCTS

ANALYSIS OF WASTE SOAP LYE

107. Determination of Total Alkali.—Transfer 10 cubic centimeters of waste lye into a clean 500-cubic-centimeter flask by means of a pipette. Dilute with 150 cubic centimeters of water, add phenol phthalein as indicator, and run in from a burette sufficient semi-normal sulphuric acid to discharge the pink color. Boil to expel carbon dioxide and titrate back with semi-normal caustic soda.

ILLUSTRATION.—*Long Method.*—Amount of $\frac{n}{2}H_2SO_4$ required for neutralization is 1.94 c. c.; 1 c. c. of $\frac{n}{2}H_2SO_4$ equals .0198 gram of $NaOH$. Total alkali, estimated as $NaOH$, equals

$$\frac{1.94 \times .0198 \times 100}{10} = .384 \text{ gram of } NaOH \text{ in 100 c. c. of liquor.}$$

Short Method.—

Total amount of $\frac{n}{2}H_2SO_4$ used is.....	5.90 c. c.
Amount of $\frac{n}{2}NaOH$ used for titrating excess of $\frac{n}{2}H_2SO_4$ is	8.96 c. c.
Amount of $\frac{n}{2}H_2SO_4$ absorbed.....	1.94 c. c.

Total alkali, estimated as $NaOH$, is

$$1.94 \times .198 = .384 \text{ gram of } NaOH \text{ in 100 c. c. of liquor.}$$

It will be observed that this gives the percentage of alkali by volume; the percentage by weight would be slightly less.

108. Determination of Sodium Chloride.—Transfer 5 cubic centimeters of waste lye into a 100-cubic centimeter beaker by means of a pipette and dilute with 50 cubic centimeters of distilled water. Mix well and transfer 5 cubic centimeters of the diluted lye to a 4-inch porcelain evaporating dish. Neutralize with dilute nitric acid and titrate with deci-normal silver-nitrate solution, using potassium chromate as indicator. Calculate the percentage of sodium chloride as though 5 grams of lye had been taken for analysis.

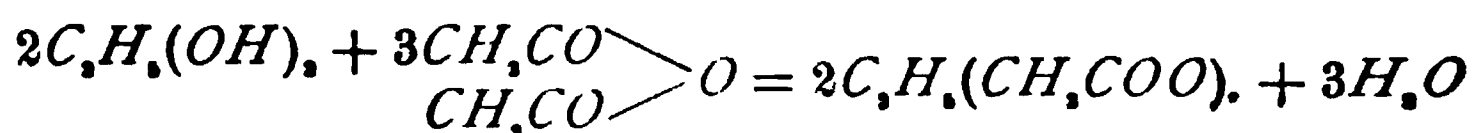
ILLUSTRATION.—Amount of waste lye taken is 5.0 c. c.; amount of $\frac{n}{10}AgNO_3$ required is 20.5 c. c.

$$\frac{20.5 \times .005868 \times 100}{5} = 2.60 \text{ per cent. of } NaCl \text{ (by volume).}$$

The true percentage by weight is found by dividing the above result by the specific gravity of the waste lye.

109. Determination of Glycerine.—As the glycerine determination requires some time, it is only done when special information as to the glycerine content of a particular lye is desired. The analytical methods used industrially are based upon the complete oxidation of glycerol to carbon dioxide and water. Potassium permanganate in either acid or alkaline solution may be used according to two methods as the oxidizing agent. In another method, potassium bichromate and sulphuric acid are used to effect

the oxidation. In another, the *acetin process*, the quantitative determination of the glycerol is based upon its transformation into glyceryl triacetin in accordance with the following equation :



This method, when carried out by a chemist skilled in its operation, gives the most accurate results. One great source of error in all the oxidation methods for the determination of glycerol is the danger of oxidizing organic impurities incompletely removed from the solution. As a result, all oxidation methods give uniformly high results. In the acetin method only the glyceride is susceptible to the action of the acetic anhydride. The method, however, is time-consuming and is not a satisfactory industrial-laboratory method. It is well to become familiar with it, as its chief value lies in its being used as a check upon the more easily and quickly manipulated oxidation methods. Again, the acetin method is not applicable to waste soap lye, because of the danger of hydrolysis of the unstable glyceryl triacetin in the presence of a large excess of water. It is used almost exclusively on crude glycerines.

110. Acetin Method.—About 1½ grams of crude glycerine are heated with 7 or 8 grams of acetic anhydride and 3 grams of anhydrous sodium acetate. The function of the last reagent is to absorb water. The mixture is gently boiled for 1½ hours in a flask provided with a water-cooled, return-flow condenser. At the expiration of this period cool the contents of the flask and introduce 50 cubic centimeters of water through the condenser. With the condenser attached, agitate with slight warming until the oily matter in the bottom of the flask has dissolved. When cool filter, and to filtrate add a few drops of phenol phthalein. Run in dilute caustic soda (20 : 1,000) from a burette until just neutral (until the yellow color just changes to reddish yellow). Now run in from a burette a measured quantity of 1 : 10 caustic-soda solution of known strength (about twice normal

or a little stronger). Heat contents of flask on water bath and titrate the excess of caustic soda with normal hydrochloric acid. The calculation of the percentage of glycerol present from the amount of sodium hydrate used to saponify the triacetin will be evident from the following calculation:

ILLUSTRATION.—The amount of (1 : 10) sodium-hydrate solution added after neutralization was 27.15 cubic centimeters. 1 cubic centimeter of (1 : 10) sodium-hydrate solution equals 2.42 cubic centimeters of normal hydrochloric acid. Therefore, 27.15 cubic centimeters \times 2.42 = 65.70 cubic centimeters, the equivalent of 27.15 cubic centimeters of (1 : 10) sodium hydrate in normal hydrochloric acid.

After saponification, 33.2 cubic centimeters of normal hydrochloric acid were required to neutralize the excess of (1 : 10) sodium hydrate. 65.7 cubic centimeters less 33.2 cubic centimeters equals 32.5 cubic centimeters of normal hydrochloric acid, which is equivalent to the sodium hydrate required for the saponification of the triacetin. 1 cubic centimeter of normal acid equals .03066 gram of glycerol; 32.5 cubic centimeters of normal acid equals .99645 gram of glycerol. Weight taken for analysis equals 1.2501 grams.

$$\frac{.99645 \times 100}{1.2501} = 79.71 \text{ per cent. glycerol present.}$$

111. Bichromate Oxidation Method.—One part of glycerol is completely oxidized to carbon dioxide by 7.486 parts of potassium bichromate in the presence of sulphuric acid. In carrying out this method, the following solutions will be necessary.

Standard Potassium Bichromate.—Dissolve 74.86 grams of pure potassium bichromate in distilled water, add 150 cubic centimeters of concentrated sulphuric acid, and when cold make up to 1 liter. This solution is of such a strength that 1 cubic centimeter equals .01 gram of glycerol. (For some purposes it is well to have a solution of one-tenth this strength made by making 100 cubic centimeters of the strong solution up to 1 liter.)

Ferrous Ammonium-Sulphate Solution.—Dissolve 240 grams of pure ferrous ammonium sulphate in 50 cubic centimeters of concentrated sulphuric acid and sufficient water,

and when cold dilute to 1 liter. Its strength should from time to time be accurately determined against the bichromate solution. Potassium ferricyanide is used as indicator on a spot plate.

Method.—Weigh out 1.5 grams of the soap lye into a 25-cubic centimeter graduated flask. For the removal of chlorine and aldehydic compounds 1.5 grams of silver oxide is added. This should be a sufficient excess. The silver oxide is acidulated with sulphuric acid. The mixture must be of uniform consistency—about that of mush. Make up to mark with distilled water and then carefully add an excess of water corresponding to the volume of the silver oxide in a dry state. Thoroughly agitate the contents of the flask until all turbidity remaining after a few seconds has disappeared. If the solution remains colored after removal of turbidity, it is due to imperfectly removed organic matter. Unless the waste lye is extremely foul, agitation with silver oxide usually suffices for complete purification. With very foul samples, basic-lead-acetate solution may be used for purification. This is added before making up to mark.

Filter through a dry filter and digest 5 cubic centimeters of this filtrate for 2 hours in boiling water with 8 to 10 cubic centimeters of the strong standard bichromate solution (accurately measured), and 15 cubic centimeters of strong sulphuric acid, in a clean covered beaker.

Titrate the excess of bichromate with the ferrous ammonium sulphate solution. Care should be taken in titrating that the standard solutions are at the normal temperature, as, being strong, a slight change in temperature makes an appreciable difference.

Subtract the excess of bichromate found from the total volume taken, the difference is the amount consumed in the oxidation of the glycerol in one-fifth of the sample. This difference in cubic centimeters multiplied by $5 \times .01 \times 100$, and the product divided by 1.5 (the weight of the sample taken) gives the percentage of glycerol in the waste lye.

SOAP ANALYSIS AND THE INTERPRETATION OF RESULTS

112. In *Quantitative Analysis* is given an exposition of the essential principles of soap analysis. As further discussion of analytical methods is unnecessary at this point, we will confine our attention to the interpretation of the results obtained by the chemist. The analysis reported is as follows:

Water.....	19.26 %
Alkali combined as soap, Na_2O	8.57 %
Alkali free as $NaOH$20 %
Alkali as Na_2CO_320 %
Fatty anhydrides.....	52.32 %
Rosin.....	19.45 %
<hr/>	
Total.....	100.00 %

We will arrange these data a little differently, so as to enable the works manager to see at a glance the information that he desires. The analysis reported is modified as follows:

Water.....	19.26 %		
Soap. (anhy- drous.)	Combined alkali, Na_2O	8.57 %	} 80.34 %
	Fatty anhydrides	52.32 %	
	Rosin	19.45 %	
	Free alkali $NaOH$20 %	
	Carbonated alkali Na_2CO_320 %	
<hr/>			
Total.....	100.00 %		

113. We will assume that the sample analyzed is a fairly fresh piece of soap, not having undergone much drying. We know that settled rosin soap fresh from the cutting table contains from 30 to 35 per cent. of water. The exceptionally small percentages of free and carbonated alkali, as well as the complete absence of even traces of unsaponifiable matter, indicate a completeness of saponification seldom arrived at in practical work. We can tell from the

appearance of the sample whether it is a cold-process or settled soap. The absence of glycerine and the practical absence of free alkali and unsaponified matter confirm our decision as to its being a settled soap. But the soap contains such a small percentage of water. It is not the ordinary settled soap with which we are familiar in this country. If it were even a straight semi-boiled soap, it would contain some glycerine. We arrive at the conclusion that the sample under examination is a settled soap that has either been grained strongly after the strengthening change—which we can tell from the completeness of saponification has been thorough—or has been subjected to a partial boiling down on closed steam, whereby, by either procedure, 10 per cent. of water has been eliminated. This soap does not contain a particle of filling of any description, from which we may infer that the liquid soap may have been pumped directly from the kettle to the frame without crutching. If the soap have a broad, open texture resulting from the slow crystallization of the sodium stearate in a menstruum of sodium oleate and sodium resinate, we are safe in saying that the soap has not been crutched. If the soap is homogeneous in texture, with no marked crystallization, the soap has been crutched, in which process the texture arising from the slow cooling of the unfilled soap in the frames is broken up by the intimate mixing in the crutcher of the soap formed from fatty acids of different melting points.

114. The chemist's report shows the presence of 19.45 per cent. of rosin and 52.32 per cent. of fatty anhydrides. From these figures we can readily arrive at the proportions of the glyceride stock and rosin that have been killed in the kettle. For purposes of calculation, we will assume the presence of 52.32 per cent. of stearic anhydride. Ninety-seven parts of stearic anhydride will yield by the absorption of the elements of water one hundred parts of stearic acid. Ninety parts of stearic acid are equivalent to one hundred parts of stearin. These equivalents become clear on working out the following proportions:

Molecular weight of stearic anhydride.....	550
Molecular weight of stearic acid.....	284
Molecular weight of stearin.....	890
Molecular weight of $3H_2O$	54
Molecular weight of glycerine.....	92

$$890 : 92 = 100 : x.$$

$x = 10.337$ per cent. glycerine yielded on saponification.

$$890 : (3 \times 284) = 100 : x.$$

$x = 95.72$ per cent. fatty acid yielded on saponification.

$$890 : 54 = 100 : x.$$

$x = 6.07$ per cent. water absorbed on saponification.

Stearin.....	100.00 parts
Glycerine formed therefrom on saponification.	10.34 parts
Fatty anhydrides yielded (approx. 90)....	89.66 parts

$$(2 \times 284) : 550 = 100 : x.$$

$x = 97$ (approx.) parts of stearic anhydride yielded by 100 parts of stearin.

52.32 per cent. stearic anhydride $\div .97 = 53.94$ per cent. stearic acid.

53.94 per cent. stearic acid $\div .9 = 59.94$ per cent. stearin.

For every 59.94 parts of glyceride stock, 19.45 parts of rosin has been used.

$$19.45 \div .5994 = 32.45 \text{ per cent. rosin.}$$

This sample is practically a 50-per-cent. rosin soap, or $33\frac{1}{3}$ pounds of rosin has been used for every 100 of glyceride stock. This is an exceptionally good soap for all laundry purposes.

CHEMICAL EXAMINATION OF REFINED GLYCERINE

115. Determination of Salt.—Fifty grams of the sample is weighed on the coarse balance into a bowl-shaped nickel crucible and heated carefully to the point of ignition. Combustion of the glycerine is allowed to proceed until the

flame goes out of itself. The carbonaceous matter remaining is carefully ignited so as not to lose any sodium chloride by volatilization or decrepitation. Dissolve the ignited mass in warm water and transfer the solution to a small porcelain evaporating dish. Titrate with deci-normal silver-nitrate solution and calculate the results.

116. Determination of Carbonaceous Matter.—Weigh 5 grams accurately into a small porcelain crucible. Heat crucible carefully to point of ignition of the glycerine and allow combustion to proceed. When complete, gently flash the flame of a Bunsen burner below the crucible to burn traces of glycerine remaining. Do not heat enough to oxidize the carbonaceous matter. Cool and weigh. The weight of the residue multiplied by 100 and divided by the weight of the sample taken gives the percentage of carbonaceous matter.

117. Determination of Ash.—Weigh out 25 grams of glycerine on the coarse balance into a weighed platinum dish. Heat carefully to ignition and allow combustion to proceed. Expel all carbonaceous matter with a strong Bunsen-burner flame, taking care to lose no salt by volatilization or decrepitation. Cool and weigh on the fine balance. Calculate the percentage of ash as in the preceding case.

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